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Love Canal Emergency Declaration Area Habitability Study. Volume 4 Soil Assessment: 2,3,7,8-TCDD (Tetrachlorodibenzo-p-Dioxin)

CH2M Hill Southeast, Inc., Reston, VA

Prepared for

Environmental Protection Agency, New York

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LOVE CANAL EMERGENCY DECLARATION AREA HABITABILITY STUDY

FINAL REPORT

VOLUME IV

Soil Assessment--
2,3,7,8-TCDD

TECHNICAL REVIEW COMMITTEE

U.S. Environmental Protection Agency Region II
U.S. Department of Health and Human Services / Centers for Disease Control
New York State Department of Health
New York State Department of Environmental Conservation

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CH2M Hill Southeast, Inc.

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9. Performing Organization Name and Address
CH2M Hill Southeast, Inc.
P. O. Box 4400
Reston, VA 22090

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Environmental studies were conducted to provide data that could be used by the Commissioner of Health for the State of New York in determining whether the Emergency Declaration Area (EDA) surrounding the Love Canal hazardous waste site is habitable. A soil assessment for 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) was conducted to determine whether the soil in the EDA contains 2,3,7,8-TCDD at levels above the level of concern set by the Centers for Disease Control: 1.0 ppb. Samples of surface soil were collected at 2,274 locations throughout the EDA.

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Prepared for

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26 Federal Plaza
New York, New York 10278

Prepared by

***CH2M HILL* SOUTHEAST, Inc.**
P.O. Box 4400
Reston, Virginia 22090

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Additional acknowledgement goes, in chronological order of their involvement, to the following organizations: the Department of Health and Human Services/Agency for Toxic Substances and Disease Registry for guidance during the design of the sampling plan; the NYSDOH for obtaining property owner permissions and scheduling the soil sampling; Cambridge Analytical Associates, Inc., for laboratory services supporting the field sampling; Ecology and Environment, Inc., for field sampling and related logistical support; Black & Veatch, Inc., for field sampling; Horizon Systems Corporation for sample tracking and automated data management; the University of Nevada at Las Vegas for sample homogenization in the laboratory; the National Enforcement Investigation Center and its contractor, Techlaw, Inc., for conducting evidentiary audits of the field sampling and sample homogenization efforts; the EPA Region II Sample Management Office and Contract Laboratory Program laboratories used for sample analysis; and the EPA Region II Environmental Services Division for data validation.

LIST OF ACRONYMS AND ABBREVIATIONS

CDC	Centers for Disease Control
CLP	Contract Laboratory Program
DHHS	U.S. Department of Health and Human Services
EDA	Love Canal Emergency Declaration Area
EPA	U.S. Environmental Protection Agency
ESD	U.S. EPA Region II Environmental Services Division
LCIC	Love Canal Indicator Chemical
MPC	Maximum possible concentration
NEIC	EPA National Enforcement Investigation Center
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OTA	U.S. Congressional Office of Technology Assessment
ppb	Parts per billion
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
SMO	U.S. EPA Region II Sample Management Office
TCDD	Tetrachlorodibenzo-p-dioxin
TRC	Technical Review Committee

1.0 SUMMARY

The *Love Canal Emergency Declaration Area; Proposed Habitability Criteria* (NYSDOH and DHHS/CDC, 1986) calls for three environmental studies. This report describes the soil assessment for 2,3,7,8-TCDD (2,3,7,8-tetrachlorodibenzo-p-dioxin, also referred to in this document as dioxin). Two other reports describe the air assessment and the soil assessment for indicator chemicals.

The habitability criteria document states that known and applicable guidelines and standards will be used where applicable to assess habitability. The known guideline for dioxin is the Centers for Disease Control (CDC) level of concern of 1.0 part per billion (ppb) dioxin in residential surface soil.

The goal of the soil assessment for dioxin was to determine whether 2,3,7,8-TCDD is present in the soil of the Love Canal Emergency Declaration Area (EDA) at concentrations of 1.0 ppb or more. Sampling point density was based on an analysis of the median size and shape of residential lots in the EDA. A 69-foot-square sampling grid was developed in order to attain a high probability of detecting a median-size contaminated lot. Specific protocols were developed to select an alternative sampling point in the event that a grid node fell on an area that could not be sampled. In addition, as a response to public request, the Technical Review Committee (TRC), which oversees the habitability assessment program, decided that a minimum of two samples should be collected on each residential lot.

Samples were collected during November and December of 1986 and during May and July of 1987. A total of 2,274 field samples were collected, and 593 quality control (QC) samples were prepared. Sampling locations (grid nodes, alternative sampling points, and two-per-lot points) were surveyed for precise identification of the sample site. The surface (2-inch depth) soil samples were homogenized and sent to an analytical laboratory. The analytical laboratories used were those in the U.S. Environmental Protection Agency's (EPA) Contract Laboratory Program (CLP). The U.S. EPA Region II Environmental Services Division (ESD) conducted validation of the analytical data.

Over and above the 2,274 points sampled, 111 planned sampling points were not sampled because of physical obstructions in the field or because of lack of permission from property owners. A total of 14 samples (0.6 percent) were not successfully analyzed during the course of the study because the analysis did not meet QC criteria and in one instance, the sample contained too much moisture.

A quality assurance (QA)/QC program was designed and implemented to monitor and maintain the quality of the study relative to stated data quality objectives. Results of the analyses of QC samples indicated that contamination had not occurred during shipping and storage. Valid results were obtained for samples representing 99.4 percent of the sampled points. The QA objectives for the study were met.

Dioxin was found at concentrations above the study target of 1.0 ppb at only one sample location (0.04 percent of the sample locations). This sample of surface soil was taken from a vacant lot in Neighborhood 4, and five aliquots were analyzed as discrete samples. Dioxin was measured at concentrations ranging from 17.3 to 21.2 ppb. Dioxin was not detected at adjacent sampling points, indicating that contiguous dioxin contamination at levels above the 1.0-ppb level of concern may not be widespread. Access to the area surrounding this sampling point has been restricted by a security fence. Further sampling of this area will be conducted under the Love Canal remedial program to identify the extent of contamination and to plan remedial activities.

Dioxin was detected at 48 sample locations (2.1 percent of the sample locations) at concentrations below the study target of 1.0 ppb. Analytical results for the remaining 2,211 sample locations (97 percent) did not contain detectable levels of dioxin.

2.0 INTRODUCTION

This report is Volume IV of a five-volume series. Volume I provides an introduction and documentation of the decision-making during the development of the Love Canal EDA Habitability Study. Volume II reports on the air assessment for indicator chemicals. Volume III presents the results of the soil assessment for indicator chemicals, and Volume V summarizes the subsequent peer review of Volumes II, III, and IV and the responses to that peer review.

This document summarizes the design and results of the soil assessment for dioxin. It is presented in six main sections and contains three appendices. Sections 3.0, 4.0, and 5.0 discuss the study goals, design, and methods, respectively. Section 6.0 summarizes the study results. The appendices contain reports from independent agencies summarizing the results of the field and laboratory audits and the analytical data validation efforts maintained throughout the study.

2.1 BACKGROUND

A detailed background of the history of Love Canal is presented in Volume I. This section provides a brief summary to assist the reader in understanding the overall intent of the Love Canal EDA Habitability Study and the role of the soil assessment for dioxin.

The former Love Canal landfill was a rectangular, 16-acre tract of land located in the southeastern end of the City of Niagara Falls in Niagara County on the western edge of New York State. The landfill takes its name from William T. Love, whose plan in the 1890s was to dig a canal between the upper and lower Niagara River to provide inexpensive hydroelectric power for a proposed model industrial city. The model city project and the partially dug Canal were abandoned before the turn of the century. The abandoned Canal was used as a chemical waste dump from 1942 to 1953. In 1953, the site was closed. Subsequently, a number of homes were built around the Canal, and the 99th Street School was built adjacent to the Canal. Soil from

the Canal was moved to the 93rd Street School site where it was used as fill. (Canal soil may also have been moved to other locations within the EDA.)

In the spring of 1978, following a series of complaints by local residents, studies were initiated to investigate the health and environmental problems at Love Canal. Under the direction of the New York State Department of Health (NYSDOH), basement sump pits were sampled and analyzed, and, under the direction of the U.S. EPA, air samples were taken at homes abutting the Canal. These results revealed significant contamination, and in August 1978, the first of two states of emergency was declared by President Carter. Homes were evacuated and remedial efforts were underway by October 1978.

Remediation at Love Canal has been extensive. Initial remediation concentrated on site containment. A clay cap was installed over the Canal area, and perimeter drains were installed during the period from May to November 1979. A leachate collection system and a leachate treatment plant were constructed during late 1979. Since that time, an extensive program for removing dioxin-contaminated sediments from area sewers and creeks has been underway. (Volume I of this report provides additional details on Love Canal remediation efforts.)

Meanwhile, concern persisted about the habitability of the residential area surrounding the Canal. In May 1980, President Carter issued a second emergency declaration for Love Canal establishing the EDA as shown in Figure 2-1, and more homes were evacuated. Eligible properties were purchased from evacuating residents by the Love Canal Area Revitalization Agency.

In the summer of 1982, the EPA released a report assessing the extent of air, water, and soil contamination in the EDA. The report was intended for use as a basis for making recommendations regarding future use of the area (U.S. EPA, 1982). Later, the U.S. Department of Health and Human Services (DHHS) reviewed the EPA study and other data to determine whether the EDA was habitable. In July 1982, after considering comments by the National Bureau of Standards on the procedures the EPA used and after further consultation with the EPA, DHHS affirmed an earlier provisional decision that the EDA was as habitable as the areas to which it had been compared. This decision was contingent on the provision that the storm sewers and their drainage tracts be cleaned and that special plans be made to perpetually safeguard against future leakage from the Canal.

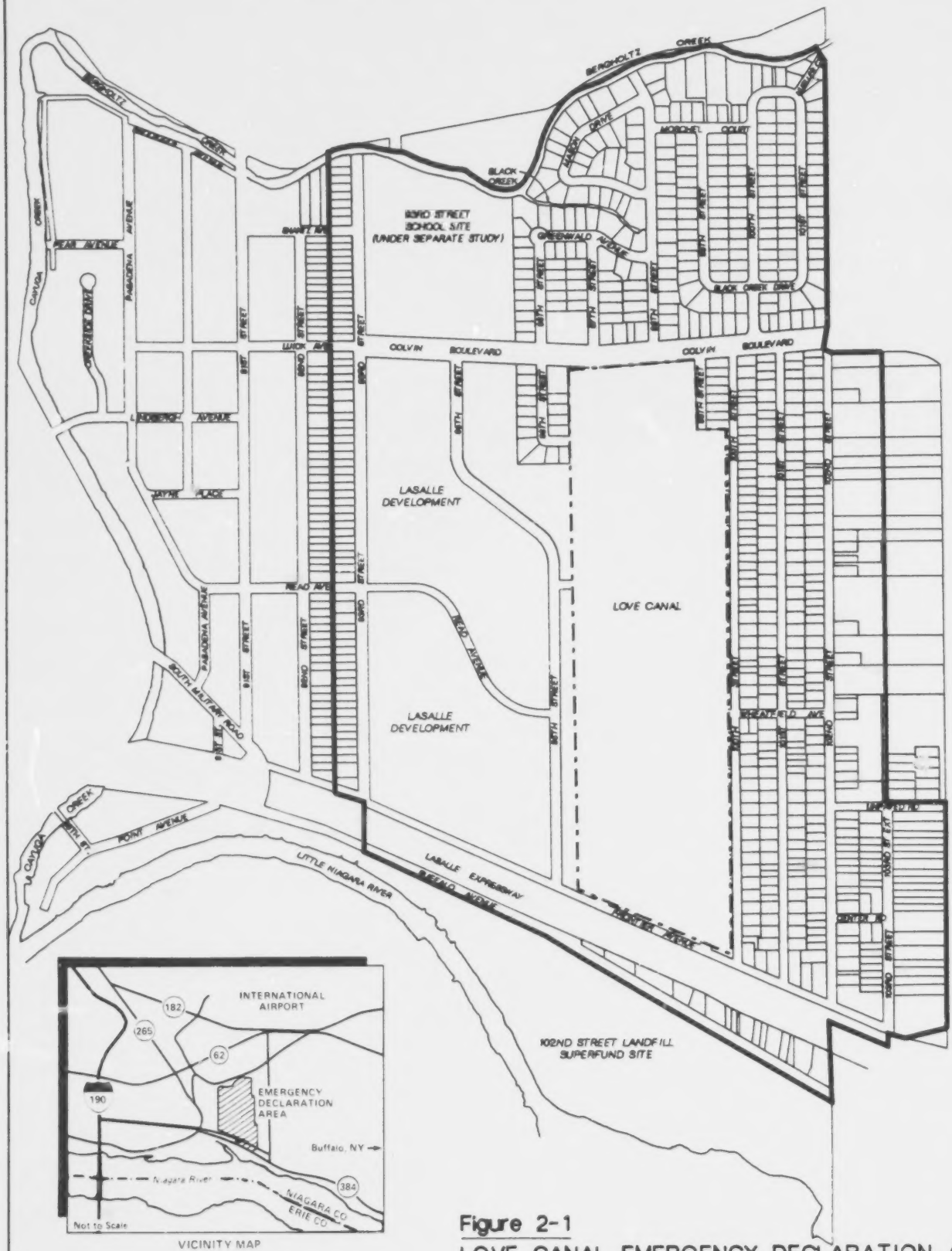


Figure 2-1

LOVE CANAL EMERGENCY DECLARATION AREA

NIAGARA FALLS, NEW YORK SCALE: 1"=750'

SOURCE:
EDA BOUNDARIES TAKEN FROM NEW YORK STATE REAL PROPERTY TAX LAW ARTICLE 17, SECTION 1702

LEGEND
EMERGENCY DECLARATION AREA (EDA) BOUNDARY

In December 1982, the U.S. Congressional Office of Technology Assessment (OTA) was requested to examine the technical basis for and validity of the habitability decision for the EDA. In June 1983, the OTA reported that, with the information available, it was not possible to conclude whether or not unsafe levels of toxic contamination existed in the EDA and that the analysis of available data did not support the DHHS decision that the EDA was as habitable as the areas with which it had been compared (OTA, 1983).

In August 1983, in response to the OTA report, the EPA established a TRC composed of representatives from the EPA, NYSDOH, DHHS/CDC, and New York State Department of Environmental Conservation to coordinate and oversee the habitability study and remedial program at Love Canal. The member agencies of the TRC asked NYSDOH and DHHS/CDC to develop criteria that would be considered by the New York State Commissioner of Health in his determination of whether or not the EDA is habitable. In 1986, the *Love Canal Emergency Declaration Area; Proposed Habitability Criteria* (NYSDOH and DHHS/CDC, 1986) was issued. The habitability criteria document underwent peer review (Life Systems, 1986) and reflects the concerns of the peer review panel.

2.2 HABITABILITY CRITERIA

The habitability criteria (NYSDOH and DHHS/CDC, 1986) stipulate that for chemicals identified in environmental media to which residents and potential residents may have significant exposure, relevant Federal or New York State standards, criteria, and guidelines will be used to assess the habitability of the EDA.

Where relevant standards, criteria, or guidelines are not available, a comparison methodology will be used to assess the relative habitability of the EDA. The habitability criteria call for a comparison of the results of the residential soil sampling and air sampling for Love Canal Indicator Chemicals (LCICs) in the EDA with the results of sampling for LCICs in similar inhabited communities in western New York State. Neighborhoods within the EDA were delineated during the development of the habitability criteria to facilitate the comparisons and habitability decision on a neighborhood basis.

The only known guideline for 2,3,7,8-TCDD is the CDC level of concern of 1.0 ppb in residential surface soil. As stated in the habitability criteria (NYS-DOH and DHHS/CDC, 1986), "A review of all federal and New York State standards, criteria or guidelines for chemicals in the Love Canal and the EDA indicates that a relevant and applicable standard exists only for TCDD (2,3,7,8 tetrachlorodibenzo-p-dioxin) in residential soils at this time." Since a guideline is available for dioxin, comparison sampling was deemed unnecessary for the soil assessment for dioxin. Therefore, according to the habitability criteria, one of the conditions for a neighborhood in the EDA to be considered habitable is that soil sample measurements of 2,3,7,8-TCDD be less than 1 ppb.

3.0 GOALS

The overall goal of the soil assessment for dioxin was to determine whether 2,3,7,8-TCDD is present at concentrations of 1.0 ppb or more in the EDA as specified in the *Love Canal Emergency Declaration Area; Proposed Habitability Criteria Document* (NYSDOH and DHHS/CDC, 1986).

At the direction of the TRC, a surface soil sampling plan was designed to have a high probability of detecting an area of contamination approximately the size of a median-size lot. A 69-foot-square sampling grid was developed based on an analysis of the median size and shape of residential lots in the EDA. Additionally, the TRC decided that a minimum of two surface soil samples should be collected per residential lot. Decisions regarding relocation of sampling points from the design location were also set forth.

The habitability criteria document (NYSDOH and DHHS/CDC, 1986) also directed that areas contaminated with levels of dioxin above 1.0 ppb were to be retested and/or remediated as appropriate. Thus, an implied operational objective of this soil assessment was to provide sufficient information to plan retesting and remediation efforts where appropriate.

Specific operational objectives of this study were to:

1. Collect samples at locations as specified by the design criteria, the directive of two samples per lot, and the relocation directives.

2. Use sample collection and analytical procedures that would identify samples with 2,3,7,8-TCDD concentrations of 1.0 ppb or more.

3. Identify and locate sample collection sites with adequate precision to allow more detailed investigations in case a value of 1.0 ppb or more was detected.

4. Meet the QA objectives of accuracy, completeness, representativeness, and comparability described in the study Quality Assurance Project Plan (QAPP) (CH2M HILL 1987, Section 4).

Sections 4.0 and 5.0, respectively, describe the study design and methods by which these objectives were attained.

4.0 DESIGN

The design of the soil assessment for dioxin was directed by the TRC and was based on the proposed habitability criteria (NYSDOH and DHHS/CDC, 1986). Generally the design of a sampling study involves three main elements: project organization, field sampling design, and QA design. Each of these three elements are discussed below.

4.1 PROJECT ORGANIZATION

Project organization and responsibilities of key personnel are detailed in this assessment's QAPP (CH2M HILL, 1986 and 1987). The assessment was conducted under the direction of EPA Region II and under the management of CH2M HILL.

The Department of Health and Human Services/Agency for Toxic Substances and Disease Registry assisted in the design of the sampling plan. The NYSDOH obtained property owner permissions and assisted in scheduling the field sampling. CH2M HILL, along with its associate firms Ecology and Environment, Inc., and Black & Veatch, Inc., conducted the field sampling. CH2M HILL's subcontractors, Cambridge Analytical Associates, Inc., and Horizon Systems Corporation, Inc., provided laboratory services and computer services, respectively, which supported the field sampling.

The University of Nevada at Las Vegas homogenized the fall 1986 samples in its laboratory. The National Enforcement Investigation Center (NEIC) through its contractor Techlaw, Inc., conducted evidentiary audits of the field sampling and sample homogenization efforts. Contract laboratories in the EPA CLP, under the direction of the EPA Region II Sample Management Office (SMO), analyzed the samples. Finally, the U.S. EPA Region II Environmental Services Division (ESD) validated the analytical data.

4.2 FIELD SAMPLING

The sampling plan for this assessment called for samples to be taken in the Love Canal EDA. (The LaSalle Expressway corridor, the Love Canal site, and the 93rd Street School site were not included in the study.) One of the most likely hypothesized modes of transport for dioxin was considered to be mechanical (e.g., movement of Canal soil for fill use). Based on this hypothesis, the target size and shape was selected to be the median size and shape of lots in the EDA. This median size and shape was analyzed statistically, and an equivalent elliptical shape and size was generated. (Elliptical shapes were preferred for statistical analyses.) The target power of the sampling plan was selected to be a 95 percent probability of detecting a contaminated area the size and shape of the equivalent ellipse (6,500 square feet with width-to-length ratio of 0.525 [Gilbert, 1987]). Using this target power, a 69-foot-square grid pattern was developed with samples to be taken at the intersection nodes.

The sampling design was based on the following assumptions:

- Dioxin contamination at the 1.0-ppb level can be reliably detected in soil samples.

- A contaminated area (greater than 1.0 ppb 2,3,7,8-TCDD) will be at least the size of the median lot in the EDA.

- Concentrations of 2,3,7,8-TCDD throughout a contaminated area will equal or exceed 1.0 ppb.

The statistical development of the sampling design is discussed in detail in the *Love Canal Dioxin Soil Sampling Quality Assurance Project Plan* (CH2M HILL, 1986, Appendix B).

The sampling scheme had to be adapted to the EDA, where approximately 25 percent of the surface is pavement, structures, densely vegetated areas, or swampy areas. The sampling grid was oriented to eliminate overlap with EDA streets running north-south and east-west, as well as with one street angled 45° west of north. The angle of rotation was selected to be an angle unlikely to cause overlap of the grid with EDA structures and/or streets. A

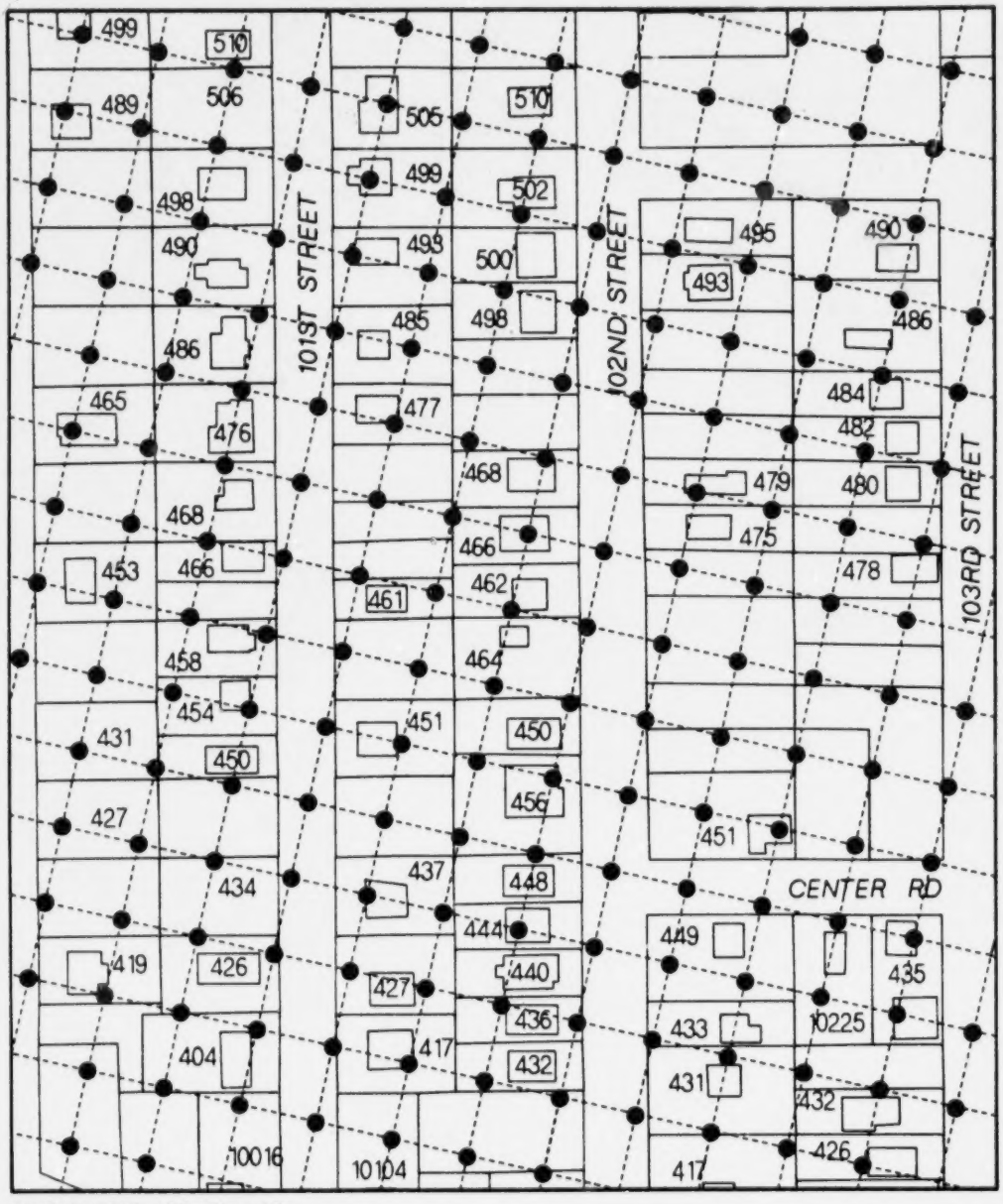
prime number of 13 was selected as the degree of rotation. An example of the grid layout is shown in Figure 4-1.

At the direction of TRC members, the sampling protocol was modified to include relocation of a sampling point within specific distance criteria when necessary. For example, in situations where nodes fell on nonsoil surfaces (e.g., structures, pavement, or swampy areas), the sampling point was relocated no less than 5 feet from any existing structures and no more than 35 feet from the node. Five feet was selected as a minimum distance from existing structures to minimize the effects of rooftop rainwater runoff. Thirty-five feet was selected as a maximum distance to relocate a sampling point from a grid node because that distance represents approximately one-half the grid block length. If those conditions could not be satisfied, the node was not used as a sampling point.

As discussed in Section 3, in addition to sampling points described by the grid system, the TRC directed that a minimum of two samples be collected in each residential lot. Sampling points to meet this criterion were incorporated into the sampling plan.

Under the direction of the TRC, the top 2 inches of soil were selected for sampling. This selection was based on a consideration of the most likely exposure mechanisms. Two common activities have been identified as a probable means by which human exposure to soil would occur: (1) children playing and (2) adults gardening, etc.

If the design could have been perfectly implemented, the 95 percent predicted probability would have been achieved. However, as indicated in the study QAPP, there are several factors that cause points to be moved or not sampled. This could lower the predicted probability; however, given the additional samples taken to provide two samples per lot, it is the opinion of the TRC that the probability of predicting the target size hot spot is as high as is reasonably achievable and meets the overall intent of the habitability criteria document as expressed in the QAPP objectives.



GRID BLOCKS ARE 69' X 69'

Figure 4-1

SOIL ASSESSMENT--2,3,7,8-TCDD
EXAMPLE GRID NODE SAMPLING PLAN

SCALE: 1"=150'

LEGEND

- STRUCTURE
- PROPERTY LINE
- 404 ADDRESS
- GRID BLOCK NODE DESIGNATING SAMPLING LOCATION

SOURCE: QAPP (CH2M HILL, 1986 and 1987C)

4.3 QUALITY ASSURANCE

As indicated in Section 3.0, Goals, the overall QA objective of the soil assessment for dioxin was to produce data of known, defensible quality, based on data accuracy, completeness, representativeness, and comparability. Accordingly, several types of QC samples were collected in the field or prepared in advance and shipped to the laboratory along with the field samples. These samples included field replicates, shipping and storage blanks, matrix spike samples, and performance evaluation samples. After the samples were received in the laboratories, all QC elements specified in the CLP "Dioxin Analysis and User's Guide" (CH2M HILL, 1986, Appendices E and F) were used. The following paragraphs describe the origin, timing, handling, analysis, and use of each type of QC sample.

Field replicates were the only QC samples collected in the field; all other QC samples were prepared in laboratories prior to field sampling. The field replicates were used to monitor variability introduced through sample handling and laboratory analysis. The field replicates were collected from a hole immediately adjacent to the last original field sample hole in each daily batch of field samples. (A batch included 16 original field samples in 1986 and 20 in 1987.) The field replicates were then treated as field samples and shipped to a laboratory along with the original field samples in that batch.

The shipping and storage blanks were samples of clean (uncontaminated) soil and were used to indicate whether contamination occurred during shipment from the equipment cleaning laboratory to the field, during storage in the field, or during shipment from the field to the laboratories. The presence of dioxin in blanks can increase uncertainty with regard to reported concentrations. If contamination was found in a shipping and storage blank, the analytical results from the samples in the associated batch would be interpreted accordingly. After a batch of original field samples was collected, the field samples were placed in the same cooler as the shipping and storage blank and shipped to the analytical laboratory. The identity of the shipping and storage blanks was not revealed to the analytical laboratory.

The matrix spike samples were used to assess overall data accuracy and to provide an indication of the efficiency of the analytical process. These matrix samples were used to monitor the extraction efficiency of the analytical system and the effects caused by the sample matrix. Routinely lower or higher

recoveries of matrix spiking compounds (i.e., dioxin) decrease the confidence in the reported values; this may indicate a serious bias toward artificially lower or higher reported values. One matrix blank was added with each batch of original field samples and sent to the analytical laboratory. The analytical laboratory spiked 1.0 ppb of 2,3,7,8- TCDD into the matrix blank and analyzed the resulting matrix spike sample along with the other samples in the same batch. The matrix spike samples were prepared according to the CLP "Dioxin Analysis Statement of Work and User's Guide" (CH2M HILL, 1986, Appendices E and F).

The performance evaluation samples were used to assess accuracy of the analytical method and the performance of each analytical laboratory. The performance evaluation samples were spiked with a known concentration of 2,3,7,8-TCDD, but the identity and concentration of the performance evaluation samples were not revealed to the analytical laboratory. The performance evaluation samples were then sent to the field where one performance evaluation sample was added to each batch of original field samples before shipment to the analytical laboratory. The performance evaluation samples were prepared and analyzed according to the CLP "Dioxin Analysis Statement of Work and User's Guide" (CH2M HILL, 1986, Appendices E and F).

5.0 METHODS

Selection of the methods used for field sample collection and analysis was based on the goal of identifying areas in the EDA that are contaminated with 2,3,7,8-TCDD at concentrations at or above 1.0 ppb. A field sample collection and homogenization method was developed to achieve this goal. The selected analytical method was the standard procedure for the analysis of the samples for 2,3,7,8-TCDD established by U.S. EPA for the CLP. Data validation was conducted to assess and control the quality of the data acquired through field activities and sample analysis.

5.1 SAMPLE COLLECTION AND HOMOGENIZATION

Surveys were conducted prior to sampling to locate the sampling points in relation to a United States Geological Survey permanent benchmark. Samples were collected by removing leaf litter and turf, taking approximately 2 inches of soil from a staked location using a stainless steel spoon, and placing the sample into an 8-ounce sample jar. The sample was then shipped to a laboratory for homogenization.

As each sample was collected, a Sample Collection Form was completed indicating the sample station number and street address along with other pertinent information. Data from the homogenization laboratory were similarly recorded on a Sample Homogenization Form. The information provided on these forms was entered into a data base. These forms are described in the study QAPP (CH2M HILL 1986 and 1987, Appendix A).

The sample homogenization process was changed at the onset of the study. The homogenization procedure adopted for the study required blending the soil sample for 90 seconds in an electric blender and then inverting and shaking the container. However, because of the physical nature of the samples (especially the high moisture and clay content), the blending process was ineffective. Therefore, all of the field samples collected during the first period of sampling (November and December 1986) were homogenized at the homogenization laboratory using a revised procedure. The revised procedure entailed hand-mixing the sample with a spoon for 90 seconds. After

homogenization, each sample was split, and each half was placed in a 4-ounce jar. One half (original sample) was sent to the analytical laboratory and one half (archive sample) to archive storage. Archive samples were kept in the event that the original sample was lost or contaminated.

Because the homogenization procedure was changed to hand-mixing, samples collected during later sampling periods (May and July 1987) were homogenized in the field. Therefore, the samples were no longer sent to the homogenization laboratory, but were sent directly to the analytical laboratory.

A complete detailed description of the sample collection, sample homogenization, and archive preparation procedures is available in the study QAPP (CH2M HILL, 1986 and 1987b).

5.2 SAMPLE ANALYSIS

All samples collected during the study were analyzed by laboratories under the direction of the EPA CLP. After homogenization, the samples were shipped to the laboratories specified by the SMO of the CLP. The analysis for dioxin was performed in accordance with the standard analytical procedures for 2,3,7,8-TCDD established by the EPA for the CLP ("Dioxin Analysis Statement of Work and User's Guide" [CH2M HILL, 1986, Appendices E and F]).

5.3 DATA VALIDATION

Validation of both field data and analytical data was conducted. The field data were assessed to determine if the sampling method and plan were properly implemented and to verify sample locations. Analytical data were validated to assess and control the quality of analytical data.

5.3.1 Field Validation

Implementation of the sample collection and homogenization methods was audited by Techlaw for the U.S. EPA NEIC. Appendix A summarizes the

findings of the evidentiary field audit performed during sample collection. Appendix B presents the report on the evidentiary audit of the homogenization laboratory. The conclusion of the audits was that both programs had met required protocols and thus had passed their respective audits.

Sample collection and homogenization records were reviewed and compared for accuracy and completeness. Since there are a large number of public and private property owners in the EDA and since two samples were required per residential lot, it was very important for sample locations to be properly tied to lot boundaries. Upon a comparison of the street address on the sample collection forms with the plotted location of the sampling points, discrepancies between lot delineations on the base map and actual field observations became evident. These sampling locations were field checked, and the Love Canal base map and data base were corrected. In addition, some points appeared to be located on the boundary of two properties. Where possible, these discrepancies were resolved using photographs taken during sample collection. If the property on which a sampling point fell could not be determined, the results were flagged with this information and both addresses given.

Implementation of the sampling plan was reviewed by an examination of the surveyor's data on sampling points that had been relocated. The distance that sampling points were moved because of field limitations (structure, pavement, dense vegetation, etc.) was calculated. The actual distance moved was compared to the criteria for movement of sampling points as set forth in the study QAPP (CH2M HILL, 1986 and 1987). For sampling points that were moved further than the criteria limit of 35 feet from the target location, a designation of "M" was incorporated following the sample station number (Example: 5280M). The original grid node was surveyed again and a new sample was collected at the node with the original sample number (Example: 5280). In each case, both samples were analyzed, and both results were reported.

5.3.2 Analytical Data Validation

Data validation was performed by the U.S. EPA Region II ESD in Edison, New Jersey. The methodology used is reported in Appendix C of this report. Analyses that did not initially pass validation were rerun to meet the QC re-

quirements called for in the CLP "Dioxin Analysis Statement of Work and User's Guide" (CH2M HILL, 1986, Appendices E and F).

6.0 RESULTS

Results of the soil assessment for 2,3,7,8-TCDD are reported below in terms of sampling activity and analytical results. Sampling activity is summarized in Section 6.1. Section 6.2 discusses the results of QA activities. The analytical findings are presented in Section 6.3, and a brief discussion of the results is given in Section 6.4.

6.1 SAMPLING ACTIVITY

Sampling activity was initiated in November of 1986, and samples were collected at 1,628 sampling points during November and December of 1986. Sampling was discontinued because of inclement weather in December 1986 and was resumed in May 1987. A total of 608 points were sampled during May 1987. Sampling activities were completed during July 1987 when samples were collected at 38 sampling points. Sixteen original field samples were collected in each daily batch during 1986, and 20 original field samples were collected in each daily batch during 1987. In addition, four QC samples were shipped with each batch.

A total of 111 of the planned sampling points could not be sampled. Forty-two grid nodes could not be sampled (or moved in accordance with the relocation criteria) because of field limitations such as dense vegetation, creek bed areas, or swampy areas. Sixty-nine grid nodes were not sampled because permission to sample could not be obtained from the property owners.

In summary, 2,274 locations were sampled during the course of the study. These points are shown in Figure 6-1. The count of each type of sample collected and/or prepared during the study is given in Table 6-1.

6.2 QUALITY ASSURANCE RESULTS

As discussed in Section 4.3, the overall QA objective of the soil assessment for dioxin was to produce data of known, defensible quality, based on the

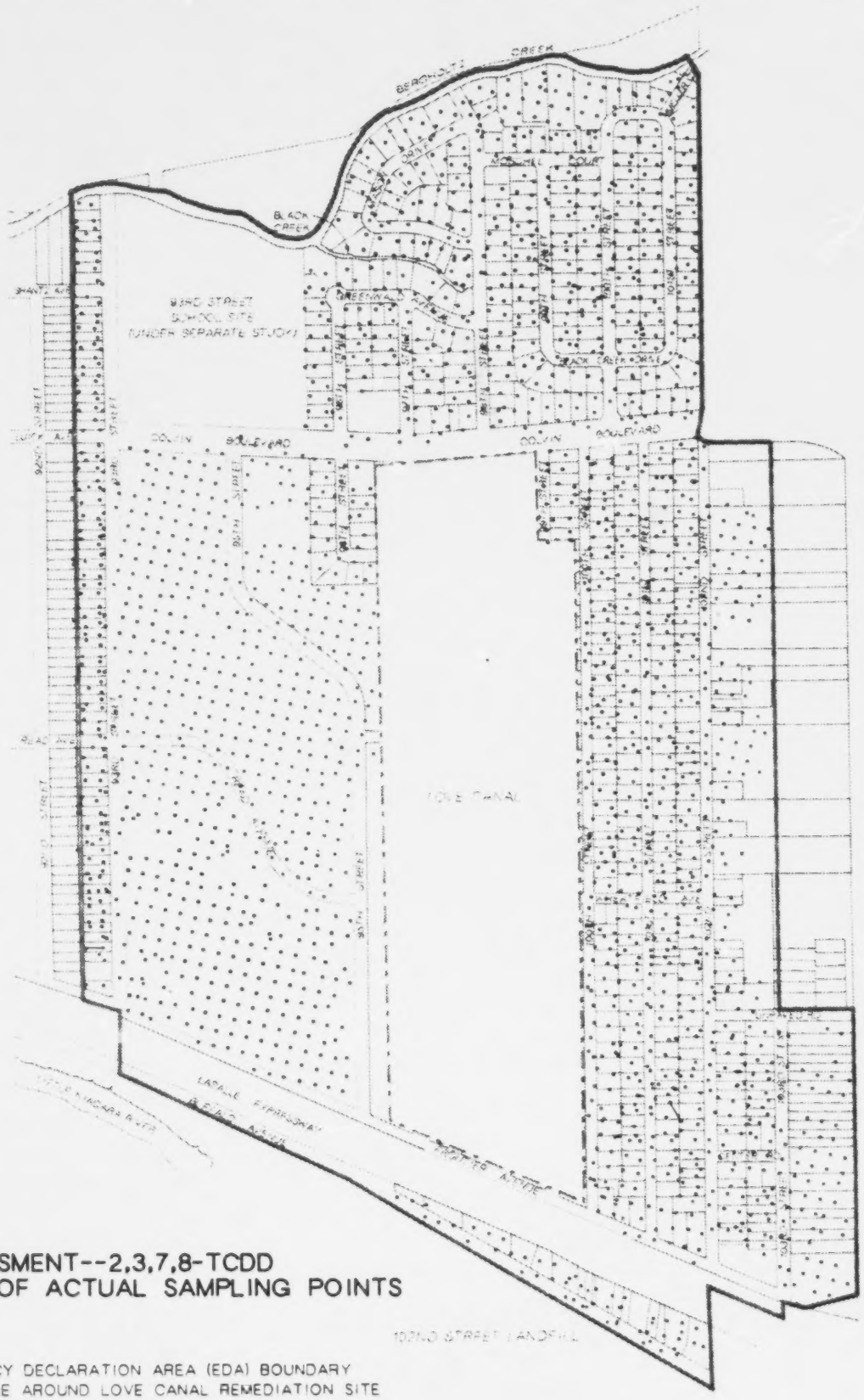


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Figure 6-1

SOIL ASSESSMENT--2,3,7,8-TCDD LOCATION OF ACTUAL SAMPLING POINTS

SCALE: 1"=650'

LEGEND

- EMERGENCY DECLARATION AREA (EDA) BOUNDARY
- FENCE LINE AROUND LOVE CANAL REMEDIATION SITE
- SAMPLING POINT

SOURCE: EDA BOUNDARIES TAKEN FROM NEW YORK STATE PROPERTY TAX LAW ARTICLE 17, SECTION 1702

Table 6-1

NUMBER AND TYPE OF SAMPLES GENERATED

Sample Type	Number Prepared	
Original field samples (taken at field sampling points)		2,274
Quality control samples (samples prepared for quality control of shipping, storage, sampling, and analysis):		
Performance evaluation sample (prepared at laboratory prior to shipment to the field)	152	
Shipping and storage blank (prepared at laboratory prior to shipment to the field)	152	
Matrix spike blank (prepared at laboratory prior to shipment to the field)	153	
Field replicate (sample collected in the field immediately adjacent to an original field sampling point)	136	
Subtotal – QC Samples	593	593
Archive samples (splits of original field samples and replicate samples)		2,410
Total Samples		5,277

data accuracy, completeness, representativeness, and comparability. The analytical data were validated, and the samples were reanalyzed if the analysis did not meet QC requirements. The results of the data validation efforts are summarized in Appendix C. Overall, the QA objectives for accuracy, completeness, representativeness, and comparability were met.

During the course of the study, a total of 279 archive samples were retrieved from storage and analyzed along with 48 QC samples. These archives were used for several purposes. One hundred and nine archive samples and 18 associated QC samples were analyzed to provide results for discarded original samples that had not been analyzed successfully. One hundred and seventy archives and 30 associated QC samples were sent to a new laboratory for a QC check. However, some of the corresponding original samples analyzed separately in another laboratory did not pass data validation. As a result, 101 of these 170 archives have valid results for both the original and the associated archive.

Of the 101 archive and original sample pairs with valid results, 5 archives and their associated original field samples contained detectable levels of dioxin. Ninety archives, along with their associated original field samples, had results of non-detects. The analytical results for these samples are given in Table 6-2. The relative standard deviation of the archive results versus the original results was 16.3 percent, indicating relatively good agreement. These results indicate that acceptable precision was achieved, considering that the data include the variations from the entire measurement process (e.g., sampling, transportation, preparation, calibration, extraction, and analysis). For samples that did not contain measurable dioxin concentrations for both original and archive samples, the increased method variability at the near-detection limit may increase the variability of the measurement process.

Of the 136 field replicate sample and original pairs collected, only one field replicate sample and its associated original field sample were found to contain detectable concentrations of dioxin. At Station No. 1700, analysis of the original sample yielded a result of 0.07 ppb, and analysis of the replicate yielded 0.07 ppb. No meaningful estimate of precision can be made because of the limited number of results.

Dioxin was not detected in any of the 143 shipping and storage blanks associated with a batch of valid results. Based on these results, there is no

Table 6-2

ARCHIVE SAMPLE RESULTS VS. ASSOCIATED ORIGINAL SAMPLE RESULTS

Sample Station Number	Original Sample Result (ppb)	Archive Sample Result (ppb)
3037	0.11[a]/0.16[a]	0.12
3409	0.20	0.19
5242	0.09	0.06
5254	0.09[a]	0.11
5277	0.23	0.19

[a]Rerun of original sample

ND = Non-Detect
MPC = Maximum Possible Concentration

Note: Only those original/archive pairs in which both samples have valid results indicating detectable levels of dioxin are included in this table.

indication that contamination occurred during shipping and storage of the samples.

Accuracy of the laboratory procedure was measured through an analysis of a performance evaluation sample and a matrix spike sample. A total of 143 performance evaluation samples were associated with valid results of field samples. The performance evaluation sample results were reviewed to determine the range of the results and the median result for each spike level of the samples. This information is provided in Table 6-3. Valid results were obtained for 146 matrix spike samples. Five matrix spike samples were analyzed twice to provide quality control on reruns of field samples. The spike level in each matrix spike sample was 1.0 ppb dioxin. The range of results was 0.68 to 1.4 ppb, and the median was 0.95 ppb 2,3,7,8-TCDD.

6.3 ANALYTICAL RESULTS

Of the 2,274 points sampled in the EDA, dioxin was detected at concentrations above the study target level of 1.0 ppb in samples taken at only one sampling location. Analysis of the original sample indicated the probable presence of TCDD above 1.0 ppb, but the concentration could not be quantified. Therefore, the archive sample was split into five separate samples and sent to a second laboratory for analysis. This sample was successfully analyzed five times, yielding results of 17.3, 19.9, 21.2, 20.9, and 20.2 ppb 2,3,7,8-TCDD. This sample was collected from a property on 100th Street between Wheatfield Avenue and Colvin Boulevard directly across the street from the Canal (see Figure 6-2).

A total of 2,517 valid results were obtained for the points sampled. Several sampling points have multiple results for a variety of reasons including analysis of field replicates, laboratory reruns due to suspected carryover (cross-contamination), confirmation of detects by high resolution analysis, and qualitative identification of dioxin followed by multiple confirmatory analyses of original field samples, replicates, and/or archives. In cases where no valid result was obtained for the original sample, the archive of the original sample was analyzed. A summary of the counts of multiple results is given in Table 6-4.

Table 6-3

SUMMARY OF PERFORMANCE EVALUATION RESULTS

Target Value (ppb)	Number of Valid Performance Evaluation Sample Results	Median of Reported Values (ppb)	Range of Reported Values (ppb)
0.76	1	(0.94[a])	--
0.80	63	0.85	0.41 - 1.10
1.20	51	0.87	0.64 - 2.10
1.54	34	1.40	0.83 - 2.30

[a]Only one result in this target value range

Note: Results are based on valid analyses of performance evaluation samples associated with batches of field samples which have passed validation. A total of six performance evaluation samples were analyzed twice to provide quality control on reruns of field samples.

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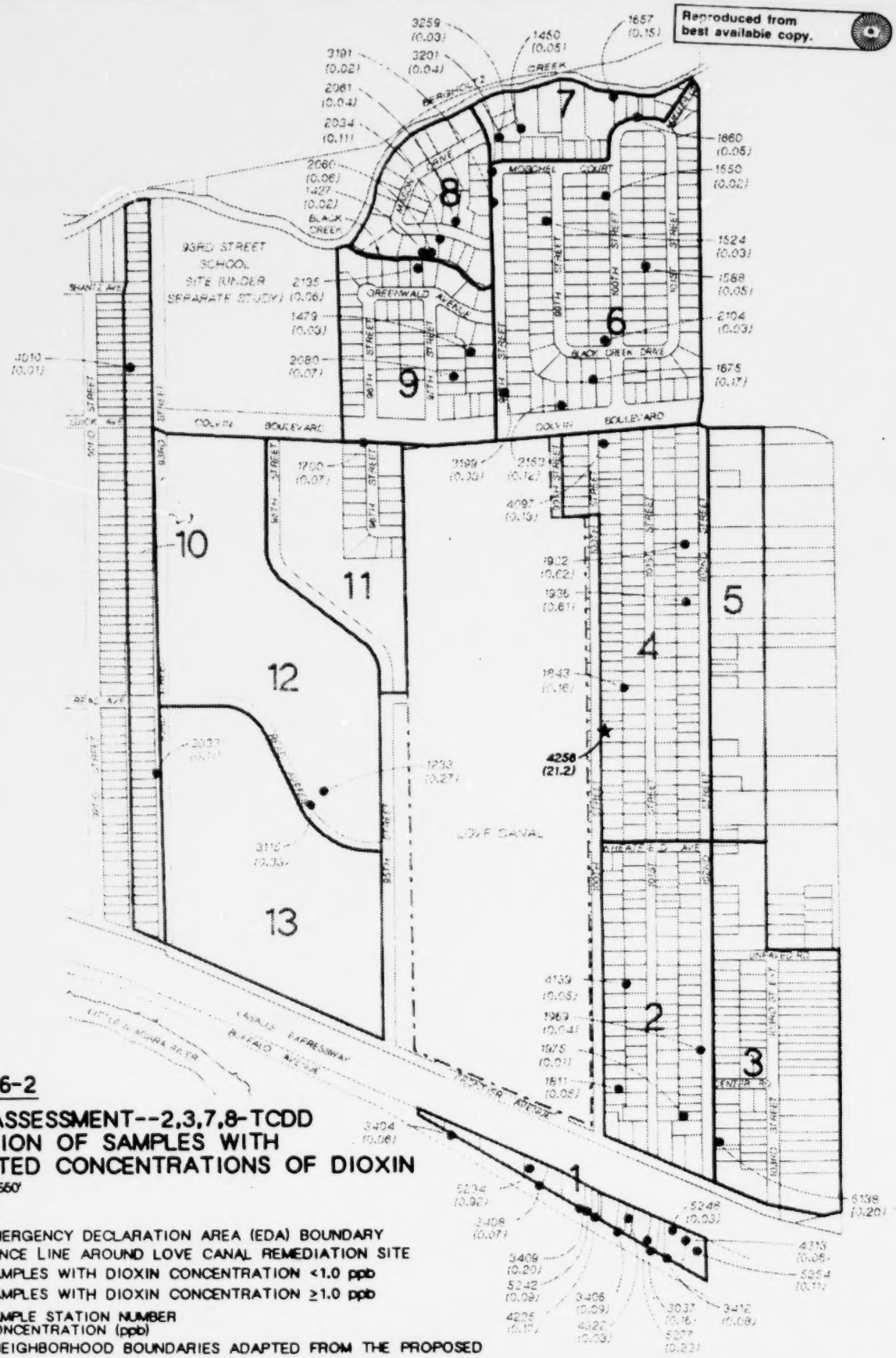


Figure 6-2

SOIL ASSESSMENT--2,3,7,8-TCDD LOCATION OF SAMPLES WITH DETECTED CONCENTRATIONS OF DIOXIN

SCALE: 1"=660'

LEGEND

- EMERGENCY DECLARATION AREA (EDA) BOUNDARY
- FENCE LINE AROUND LOVE CANAL REMEDIATION SITE
- ● SAMPLES WITH DIOXIN CONCENTRATION <1.0 ppb
- ★ SAMPLES WITH DIOXIN CONCENTRATION ≥1.0 ppb
- 4258 (21.24) SAMPLE STATION NUMBER CONCENTRATION (ppb)

SOURCE: NEIGHBORHOOD BOUNDARIES ADAPTED FROM THE PROPOSED HABITABILITY CRITERIA DOCUMENT (NYSDOH AND DHHS/CDC, 1986).

Table 6-4

DISTRIBUTION OF ANALYTICAL RESULTS WITH RESPECT TO SAMPLE LOCATION

Number of Analytical Results Reported per Sample Location	x	Number of Sample Locations	=	Number of Analytical Results[a]
0		14		0
1		2,019		2,019
2		230		460
3		7		21
4		3		12
5		1		5
Totals		2,274		2,517

[a]All analytical results included have passed data validation.

Valid analyses were not obtained for samples taken at 14 locations (0.6 percent of the points sampled). One sample was not analyzed because of excessive moisture in the sample. The remaining 13 samples were analyzed, and the results were rejected because the QC requirements were not met. Several of these samples were reanalyzed, but the results again did not meet the QC requirements. The laboratory forms for these samples and location of these sampling points were reviewed, and a decision was made not to reanalyze these samples because the completeness objective stated in the study QAPP (CH2M HILL, 1986 and 1987a) had been met.

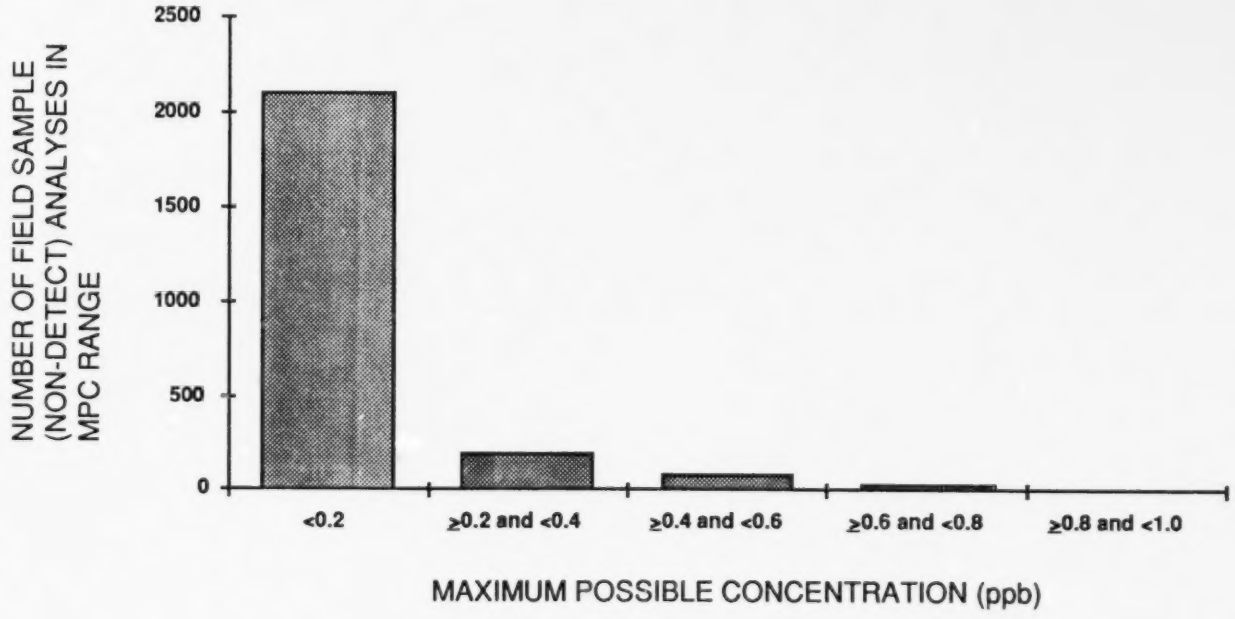
A total of 2,454 analyses (97 percent) were non-detects. The detection limit for the analysis of each sample is described as the Maximum Possible Concentration (MPC) that could go undetected in the sample. The MPC varies for each sample analysis due primarily to analytical process parameters, instrument performance, and matrix interferences. The CLP "Dioxin Analysis Statement of Work and User's Guide" (CH2M HILL, 1986, Appendices E and F) required the MPC in these analyses to be less than 1.0 ppb. Results with an MPC greater than 1.0 ppb required reanalysis in order to meet this criterion. The distribution of MPC values throughout the analysis of field samples is illustrated in Figure 6-3.

Dioxin was detected in a total of 63 analyses, representing a total of 49 sampling points (2.2 percent of the sampling points). These analytical results are listed in Table 6-5. The sampling points with detectable levels of dioxin are indicated on Figure 6-3 with the highest analytical result for each sampling point shown. The overall results of the study are summarized by concentration interval in Table 6-6.

6.4 DISCUSSION OF RESULTS

Dioxin was detected at levels above the CDC level of concern of 1.0 ppb at only one sampling point (0.04 percent) of the 2,274 points sampled. The remaining sampling points with detected concentrations of dioxin were below this level of concern.

The detection of dioxin at one sampling point at a level equal to or greater than the study target level of 1.0 ppb indicates that a contaminated area exists. Results of samples from nearby sampling points were non-detects. No



NOTE: Original field samples, replicates, and archives only.

FIGURE 6-3 SOIL ASSESSMENT–2,3,7,8-TCDD FREQUENCY OF MPC VALUES FOR ANALYSES OF FIELD SAMPLES WITH NON-DETECT RESULTS

Table 6-5

SUMMARY OF ANALYTICAL RESULTS FOR SAMPLES WITH DETECTED CONCENTRATIONS

Station Number	Type of Sample	Initial Result (ppb)	Rerun Result (ppb)	Comments
1233	Original	0.27	ND (MPC = 0.04)	
1427	Original	0.02	-	
1450	Original	N/A	0.05	
1479	Original	N/A	Rejected	
			0.03	Sample reextracted and reanalyzed
1524	Original	Rejected	-	
	Archive	0.03	-	
1550	Original	0.02		
1588	Original	ND (MPC = 0.04)	-	
	Replicate	0.05	-	
1657	Original	N/A	0.15	
1660	Original	N/A	0.05	
1675	Original	0.17	ND (MPC = 0.02)	
1700	Original	0.07	-	
	Replicate	0.07	-	
1811	Original	N/A	Rejected	
			0.08	Sample reextracted and reanalyzed
1843	Original	0.16	-	
1932	Original	0.62	-	
1936	Original	0.61	ND (MPC = 0.04)	
1969	Original	0.04	-	
1975	Original	ND (MPC = 0.05)	-	
	Archive	0.01	-	
2034	Original	0.11	-	
2060	Original	0.06	-	
2061	Original	0.04	-	
2080	Original	0.07	-	

MPC = Maximum possible concentration
ND = Not detected
N/A = Not available (lab decision to rerun sample)
Rejected = Data rejected by ESD
- = No analysis performed

Table 6-5
(Continued)

Station Number	Type of Sample	Initial Result (ppb)	Rerun Result (ppb)	Comments
2104	Original	Rejected	-	
	Replicate	Rejected	-	
	Archive	ND (MPC = 0.04)	-	
	Replicate	0.03	-	
2135	Original	0.06		
2153	Original	Rejected	0.12	
3033	Original	0.11	ND (MPC = 0.05)	
3037	Original	Rejected	0.11	
			0.16	
	Archive	0.12	-	
3115	Original	0.33	-	
3191	Original	ND (MPC = 0.08)	-	
	Replicate	ND (MPC = 0.08)	-	
	Archive	0.02	-	
3199	Original	0.03	-	
3201	Original	0.04	-	
3259	Original	0.03	-	
3404	Original	Rejected		
	Archive	0.06		
3406	Original	Rejected	-	
	Archive	0.09	-	
3408	Original	Rejected	-	
	Archive	0.07	-	
3409	Original	N/A	0.20	
	Archive	0.19	-	
3412	Original	Rejected	-	
	Archive	0.08	-	
4010	Original	N/A	Rejected	
			0.01	Sample reextracted and reanalyzed

MPC = Maximum possible concentration
ND = Not detected
N/A = Not available (lab decision to rerun sample)
Rejected = Data rejected by ESD.
- = No analysis performed

Table 6-5
(Continued)

Station Number	Type of Sample	Initial Result (ppb)	Rerun Result (ppb)	Comments
4097	Original	0.13	0.12	
	Duplicate[a]		0.10	Duplicate analysis of rerun sample
4139	Original	N/A	Rejected	
			ND (MPC = 0.04)	Sample reextracted and reanalyzed
	Replicate	N/A	Rejected	
			0.05	Sample reextracted and reanalyzed
4225	Original	Rejected	Rejected	
	Archive	0.17	-	
4256	Original	Rejected	Rejected	
	Archive	Rejected	17.3	
		Rejected	21.2	
		Rejected	Rejected	
		Rejected	20.9	
		19.9	20.2	
4313	Original	0.06	-	
4322	Original	0.03	-	
5138	Original	0.20	-	
5234	Original	Rejected	ND (MPC = 0.36)	
			ND (MPC = 0.19)	
	Archive	0.63	0.92	Utilized high resolution technique for rerun result
5242	Original	N/A	0.09	
	Archive	0.06	-	
5248	Original	N/A	0.03	
	Archive	ND (MPC = 0.04)	-	
5254	Original	Rejected	0.09	
	Archive	0.11	-	
5277	Original	N/A	0.23	
	Archive	0.18	-	

MPC = Maximum possible concentration
ND = Not detected
N/A = Not available (lab decision to rerun sample)
Rejected = Data rejected by ESD
- = No analysis performed
[a]Duplicate analyses are described in Appendix C.

Table 6-6

DISTRIBUTION OF ANALYTICAL RESULTS WITH RESPECT TO CONCENTRATION RANGES[a]

Concentration Range (ppb)	Number of Sampling Locations with Highest Result in Concentration Range	Total Number of Results Using All Reported Concentrations for Each Sampling Location[b]
ND	2,211	2,454
<0.2	40	49
≥0.2 and <0.4	5	5
≥0.4 and <0.6	0	0
≥0.6 and <0.8	2	3
≥0.8 and <1.0	1	1
≥1	1	5[c]
	2,260	2,517
Sampling locations for which analysis did not pass QC:	14	
Total number of samples collected:	2,274	

[a]All analytical results presented have passed QC criteria.
[b]It is possible that one sample location could be represented in two or more concentration ranges if multiple analytical results were reported.
[c]These validated analytical results (for sample location at Station 4256) are 17.3, 21.2, 20.9, 20.2, and 19.9 ppb 2,3,7,8-TCDD.

house is located on this lot, and the nearest detect is approximately 260 feet away, with a concentration of 0.16 ppb 2,3,7,8-TCDD. The area surrounding the hit has been fenced and secured. Further sampling will be conducted under the Love Canal remedial program to determine the extent of the contamination and to plan subsequent corrective action.

REFERENCES

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Gilbert, Richard D. *Statistical Methods for Environmental Pollution Monitoring.* Van Nostrand Reinhold Company, New York.

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U.S. EPA, Office of Research and Development. 1982. *Environmental Monitoring at Love Canal.* Volumes I, II, and III.

APPENDIX A
Field Evidentiary Audit Report

Prepared by

U.S. EPA National Enforcement Investigation Center
Techlaw, Inc.
Lakewood, Colorado

CONTRACTOR
EVIDENCE
AUDIT
TEAM

December 31, 1986

Mr. Doug Garbarini
USEPA Region II
26 Federal Plaza
Room 737
New York, NY 10278

RE: Transmittal of Field Audit Report for Love Canal Dioxin Soil Sampling

Dear Doug:

Enclosed are two copies of the field audit report for the Love Canal Dioxin Soil Sampling conducted November 17-19, 1986.

The report was received and approved by NEIC and a copy has been transmitted to Mr. Craig Rightmire of CH2M Hill as you requested.

Also enclosed is an evidence audit Work Plan for this audit.

Thank you for requesting our participation in this study and we were pleased to provide this evidence audit support on behalf of NEIC. If you have any questions, please contact us at (303) 233-1248 or Rob Laidlaw at (303) 236-5122 (FTS 776-5122).

Best wishes for the New Year.

Keith Wegner
Staff Consultant

KW:rls

Enclosures

cc: Rob Laidlaw, NEIC (w/enclosure)
Betty Malone, CEAT (w/enclosure)

IF: 222-068

6-11

FIELD AUDIT REPORT

LOVE CANAL
Dioxin Soil Sampling Study
Niagara Falls, New York

November 17-19, 1986

CH2M Hill
P.O. Box 4400
Reston, Virginia 22090
(703) 471-1441

Craig Rightmire - Field Team Manager
Sadia Kissoon - Document Control Manager
Ana Morera - Field Auditor
Don Johnson - Field Auditor

Horizon Systems Corporation
1800 Michael Faraday Drive
Suite 201
Reston, Virginia 22090
(703) 471-0480

Robert Meyer - Document Control Technician

EPA - Region II
(212) 264-0722

Douglas Garbarini - Love Canal Regional Project Manager

REACT - Denver
(303) 233-1248

Betty Malone - Field Evidence Auditor
Troy Sanders - Field Evidence Auditor

Ecology and Environment, Inc.
195 Sugg Road, P.O. Box D
Buffalo, New York 14225
(716) 633-9881

Sampling Team 1

Joe Jackson	- Field Team Supervisor
Joe Chandler	- Sample Collector, Team 1A
Michael Michalowski	- Sample Collector, Team 1A
Mary Miller	- Sample Collector, Team 1B
Jon Nickerson	- Sample Collector, Team 1B

Sampling Team 2

Russell Short	- Field Team Supervisor
Keith Keller	- Sample Collector, Team 2A
Pamela Gunther	- Sample Collector, Team 2A
M.J. Farrell	- Sample Collector, Team 2A
Thomas Ferraro	- Sample Collector, Team 2B
Thomas Siener	- Sample Collector, Team 2B

Sampling Team 3

Scott McCone	- Field Team Supervisor
Bret Maxwell	- Sample Collector, Team 3A
William Hall	- Sample Collector, Team 3A
Dale Dolph	- Sample Collector, Team 3B
Gene Florentino	- Sample Collector, Team 3B

Black and Veatch
Pritchard Building
8205 West 108th Terrace
Overland Park, KS 66210

Sampling Team 4

Richard Gibbs	- Field Team Supervisor
Teresa Shock	- Sample Collector, Team 4A
Gary Schnettgoecke	- Sample Collector, Team 4A
Jeff Dilley	- Sample Collector, Team 4B
David Munie	- Sample Collector, Team 4B

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA Contract No. 68-01-7104 for Superfund Site No. 28.

INTRODUCTION

On November 17, 18, and 19, 1986, NEIC Regional Evidence Audit Contract Team (REACT) personnel conducted an audit to observe document control and chain-of-custody procedures followed during the collection of soil samples for the Love Canal Dioxin Soil Sampling Study. An initial meeting was held in Niagara Falls, New York on November 17, to discuss sampling procedures and protocol. Present at the meeting were S. Kissoon, D. Garbarini, T. Sanders, and B. Malone. On November 18 and 19, sampling procedures and documentation were observed by REACT personnel. The CH2M Hill sampling plan was also examined, and is briefly described below:

Sampling Plan

A sampling plan (Quality Assurance Project Plan (QAPP)) was prepared for the sample survey by CH2M Hill and was reviewed and approved by the Region II EPA.

The soil sampling protocol contains:

1. Project description and background
2. Sampling procedures and custody
3. Documentation of sample collection
4. Quality Assurance Program
5. Site Safety Plans

The following documents were reviewed during the sample collection phase of the audit: Sample Collection Labels, Sample Collection Forms, Chain-of-Custody Forms, and field and photograph logbooks.

Accountable Field Documents

A tracking system is used, in which the following documents are numbered and are accountable:

1. Sample Collection Form
2. Chain-of-Custody Form for Shelby Tubes
3. Field Logbooks

Incorrectly completed forms and those forms which are not used are marked "Void" and returned to Ana Morera, a CH2M Hill field auditor. Sample collection labels are not numbered.

A brief description of each of the documents reviewed during the sample collection phase of the audit appears below. A table listing specific documents examined during the audit appears in the Appendix.

A-1

Sample Collection Labels

Sample collection labels were used in lieu of sample tags by CH2M Hill during the sampling episode. A label was completed for each sample collected. Each sample collection label contains the following information: Project title, jar number, date, time, collector, project number, client, chemical analyses, and comments.

During the period of the audit, ten Sample Collection Labels were selected at random and examined for completeness by the auditors. All sample labels were completed and were consistent.

Sample Collection Forms

Sample Collection Forms were used by CH2M Hill with one form completed for each sample collected. Each form, completed and signed by the collector, contains information concerning sampling locations, sample descriptions, shipping information, and samples associated with blanks and replicates.

During the audit, one hundred Sample Collection Forms were reviewed by the REACT auditors. The auditors noted that in forms completed by one of the sampling teams on November 18, 1986, the name of the sample collector appearing in the top portion of the form was not the same name that was signed as the collector at the bottom of the form. (REACT personnel recommended that the incorrect name be changed so that both names are in agreement. It was suggested that, as in the chain-of-custody forms, all sample collectors' names be included in the top portion of the form, allowing any one of the sample collection team members to sign at the bottom of the form.)

REACT auditors also compared information from the sample collection forms with the chain-of-custody records. The chain-of-custody forms will be discussed in the following section.

Chain-of-Custody Form

EPA-type Chain-of-Custody Record Forms were used by CH2M Hill, and were completed for all samples collected. These forms accompanied the samples from their origin at the site to the sample homogenization laboratory in Las Vegas, Nevada. Information on these forms includes: Project name and number, shipment information, sample collection information, and signatures and dates denoting when the samples were relinquished and received.

Eight completed chain-of-custody records were reviewed by the REACT auditors. The following recommendations were made:

- o the signature of the individual who initially relinquishes the sample should also appear in the "Sampled By and Title" portion of the custody record. This was correctly completed on the majority of the forms examined by REACT personnel.

- o sampling team members should be careful to enter all required information on the custody forms. The majority of the forms were fully completed; however, several were missing information such as the method of shipment, the airbill number, and complete dates.

It was suggested during the debriefing that the sample jar number from the Sample Collection Label be included under the Remarks section of the chain-of-custody form, as an added means of sample identification.

Information which appeared on both the chain-of-custody and sample collection forms was compared for consistency. It was noted that corrections made to the Sample Collection forms were not made to corresponding entries on the Chain-of-Custody Records. (REACT auditors recommended that the same changes be made to both forms to ensure consistency.)

Field Logbook

The sampling team members recorded information pertaining to the sampling effort in a field logbook. The initial page of each entry was signed and dated by the author. The logbook was project-specific for Love Canal. The logbook was numbered upon assignment and identified as an accountable document.

Information recorded in the logbook included:

- o Sampling site locations and station numbers
- o Date of sampling
- o Names of samplers
- o Weather conditions
- o Temperature
- o Deviations (i.e., broken sample bottles, equipment malfunctions)
- o Picture and roll numbers of photographs taken

REACT personnel examined logbooks completed by the eight sampling teams. CH2M Hill field auditor Ana Morera was advised that the sampling teams should be careful to sign and date each logbook page and to fill in blank spaces with diagonal slashes. It was also pointed out that logbook covers did not contain the name of the person or organization to whom the book was assigned, as specified in the CH2M Hill Quality Assurance Project Plan.

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Photograph Logbook

A separate logbook was used to retain information regarding photographs. The photograph logbook was not identified as an accountable document. Information recorded in the logbook included:

- o Date and location of sample site
- o Polaroid and 35 mm photo numbers
- o Sample station number
- o Serial number of 35 mm camera body and lens
- o Preparer

(REACT personnel recommended at the debriefing that the names of persons taking photographs be included in the logbook.)

Field Observations

Prior to the audit, a briefing was held on November 17, 1986, with the personnel listed in the introduction of this report. Topics discussed included sampling procedures, sampling documentation, the purpose of the audit, and health and safety issues.

On November 17, 18, and 19, 1986, soil samples were collected at Love Canal in Niagara Falls, New York. Eight sampling teams, consisting of personnel from Ecology & Environment and Black & Veatch, collected surface soil samples to be later analyzed for Dioxin. At the end of each day's sampling episode, all Sample Collection Forms and Chain-of-Custody forms were reviewed by the CH2M Hill field auditor. On each following day, data from the Sample Collection forms was entered by Horizon Systems Corporation into a computerized database.

Custody of the samples was maintained in the following way: During sample collection, the Field Team Supervisor (FTS) for each sampling team remained in the respective team's sampling van to complete documentation and to prepare the sample coolers for shipping. When possible, samples were shipped at the end of each day. If there were not enough samples collected in a day to completely fill a cooler, the samples were locked up overnight at the Ecology & Environment office in Buffalo, NY, and shipped the following day.

REACT personnel examined documentation for samples collected November 17 and 18, 1986. Documentation related to each day's sampling effort was reviewed the following morning subsequent to review of the documents by the CH2M Hill field auditor.

A debriefing was held on November 19, 1986. Details of the debriefing are discussed below.

Debriefing

A debriefing was held at the Love Canal site on November 19, 1986. Craig Rightmire, Douglas Garbarini, and the REACT auditors were present.

C. Rightmire was informed that the overall organization of the sampling teams in collecting samples and completing associated documentation appeared to be well conceived and generally consistent with the sampling plan.

The following recommendations were made during the debriefing:

1. Names of sample collectors appearing in the top and bottom portions of the Sample Collection Form should be in agreement.

2. The name of the person initially relinquishing the sample should also be included as one of the sampling team members in the "Sampled By and Title" portion of the Chain-of-Custody Record.

3. Care should be taken to complete all information requested on the Chain-of-Custody Record.

4. All pages of field logbooks should be initialed or signed, and dated.

5. Information contained on logbook covers, such as the name of the person or organization to whom the book was assigned, should be included, to be consistent with that specified in the Quality Assurance Project Plan.

6. The photograph logbook should contain the names of persons taking photographs.

Summary

The following procedures were observed by the REACT auditors during the sample collection phase of the audit:

1. An accountable document system was utilized for documenting sample collection.

2. Project specific logbooks were utilized in which pertinent information, such as sample locations and field observations were recorded for collection.

3. Chain-of-custody procedures were being followed and sample identity was maintained for sample collection procedures.

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4. Sample security was maintained by keeping samples under custody or stored within secured areas when not in the physical custody of the samplers.

5. The CH2M Hill Quality Assurance Project Plan was followed except where field conditions warranted deviations. Any deviations were noted in the field logbooks.

APPENDIX

RECORD OF DOCUMENTATION USED FOR DIOXIN SAMPLES COLLECTED NOVEMBER 18-19, 1986

LOVE CANAL FIELD AUDIT

Record of Samples Collected
November 18-19, 1986

Station Number	Sample Collection Form Number	Chain-of-Custody Form Number
4141	30183	1011
4140	30184	1011
3338	30177	1011
2200	30181	1011
3302	30092	1011
4262	30179	1011
1870	30100	1011
2222	30101	1011
1808	30087	1011
3300	30088	1011
4122	30091	1011
5256	30095	1011
1872	30098	1011
1809	30089	1012
4043	30090	1012
1812	30093	1012
1817	30094	1012
1815	30096	1012
5267	30097	1012
1919	30099	1012
2221	30178	1012
4263	30180	1012
1866	30182	1012
3339	30102	1012
3005	30027	1004
2007	30028	1004
1001	30030	1004
3001	30031	1004
5235	30021	1004
5249	30016	1004
4001	30033	1004
1004	30137	1004
1007	30139	1004
1013	30146	1004
2008	30141	1004
1011	30143	1004
4143	30216	1013
5251	30022	1017
3407	30020	1017
4002	30032	1017
2002	30029	1017

Station Number	Sample Collection Form Number	Chain-of-Custody Form Number
4006	30026	1017
3004	30025	1017
4300	30024	1017
5234	30023	1017
3010	30142	1017
1010	30140	1017
4005	30138	1017
4223	30034	1017
2009	30144	1017
1012	30145	1017
5248	30017	1017
4177	30211	1015
4176	30210	1015
1871	30206	1015
1868	30208	1015
1920	30204	1015
3341	30202	1015
2176	30199	1015
1813	30197	1015
2174	30195	1015
3335	30214	1015
2219	30215	1016
1918	30212	1016
4173	30217	1016
1867	30207	1016
2220	30213	1016
1867	30209	1016
1816	30205	1016
3342	30203	1016
3301	30201	1016
4121	30200	1016
1807	30196	1016
1811	30198	1016
2010	30068	1019
5253	30054	1019
5274	30056	1019
3006	30058	1019
1006	30061	1019
1008	30063	1019
3009	30065	1019
4009	30066	1019
3426	30157	1019
3003	30158	1019
2004	30160	1019
1005	30162	1019
4007	30164	1019

Station Number	Sample Collection Form Number	Chain-of-Custody Form Number
5236	30057	1020
5254	30059	1020
3007	30060	1020
4004	30062	1020
3008	30064	1020
1003	30067	1020
2005	30156	1020
2003	30159	1020
1002	30161	1020
1009	30163	1020
4008	30165	1020
4010	30166	1020
3037	30055	1020

APPENDIX B
Homogenization Laboratory Evidentiary Audit Report

Prepared by

U.S. EPA National Enforcement Investigation Center
Techlaw, Inc.
Lakewood, Colorado

CONTRACTOR
EVIDENCE
AUDIT
TEAM

January 21, 1987

Mr. Doug Garbarini
USEPA Region II
26 Federal Plaza, Room 737
New York, NY 10278

RE: Transmittal of Evidence Audit Report for UNLV-ERC
Related to Love Canal Dioxin Soil Sampling Study

Dear Doug:

Enclosed is the audit report for the evidence audit conducted at the University of Nevada, Las Vegas - Environmental Research Center facility. The report has been reviewed and approved by NEIC, and we are transmitting this copy to you.

With your concurrence we have also transmitted a copy of the report to the UNLV-ERC Laboratory.

Please contact us if you have any comments or questions.

Yours sincerely,

Keith Wegner

Keith Wegner
Staff Consultant

KW/lkl

Enclosure

cc: Rob Laidlaw, NEIC (w/enclosure)
Betty Malone, CEAT (w/enclosure)
IF: 111-068/222-068

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LABORATORY EVIDENCE AUDIT REPORT

UNIVERSITY OF NEVADA AT LAS VEGAS
QUALITY ASSURANCE LABORATORY
ENVIRONMENTAL RESEARCH CENTER

December 15, 1986

UNLV-Quality Assurance Laboratory
Environmental Research Center
4505 Maryland Parkway
Las Vegas, NV 88154
(702) 739-3142

Joseph Campana - Director, Quality Assurance Laboratory
Steve Ward - Chemist
Amy Smiecinski - Quality Assurance Specialist

CH_2M Hill - Reston, VA
(703) 471-1441

Ana Morera - Sample Custodian/Auditor

Black and Veatch - Overland Park, KS
(913) 661-6421

Patrick Dunn - Sample Preparation Technician
Rodney Rowe - Sample Preparation Technician
Michael Boehler - Sample Preparation Technician

USEPA - Region II/New York, NY
(212) 264-0722

Doug Garbarini - Love Canal Dioxin Soil Sampling
Project Manager

REACT - NEIC/Denver, CO
(303) 233-1248

Keith Wegner - Evidence Auditor/Audit Team Leader
Betty Malone - Evidence Auditor

This work was conducted on behalf of the Environmental Protection Agency's (EPA) National Enforcement Investigations Center (NEIC) under EPA contracts68-01-7104 and 68-01-7369.

An audit of laboratory operations pertaining to chain-of-custody and document control procedures was conducted at the Environmental Research Center of the University of Nevada Las Vegas Quality Assurance Laboratory (ERC-UNLV) on December 15, 1986.

Soil samples collected during the Love Canal Dioxin Soil Sampling (LCDSS) study are being prepared for analysis at ERC-UNLV under a work assignment through EPA contract 68-01-7159. The soil samples are being prepared using a team of ERC-UNLV and contractor personnel. The contractor teams and their responsibilities are described below:

- CH_2M Hill - Receive and log in samples at the ERC-UNLV facility. Sample custodian and document control responsibilities.

- Black and Veatch - Receive logged-in samples from CH_2M Hill and prepare samples for homogenization in ERC-UNLV containment laboratory. Processing, packaging, labeling, shipping, and documentation responsibilities.

- ERC-UNLV - Receive processed samples from Black and Veatch and perform actual sample homogenization in ERC-UNLV containment laboratory. Sample homogenization and tracking documentation responsibilities.

During the audit, the audit team examined the procedures and accompanying documentation related to the following: sample receiving, sample storage, sample tracking (from receipt to completion of preparation), and document organization and filing.

SAMPLE RECEIVING

Sample shipments were received by ERC-UNLV personnel. A. Morera of CH_2M Hill is currently unpacking the shipments and logging them in.

All samples received at the ERC are recorded in an ERC-UNLV Sample Receipt Logbook. This bound logbook contains the following information:

1. Shipment number
2. Airbill number
3. Date
4. Cooler number
5. Number of coolers
6. New UNLV cooler numbers
7. Condition of seals on coolers
8. Signed and dated pages

For Love Canal soil samples received under this work assignment, two additional document logs are completed. The

first log is titled LCDSS Homogenization and Unpackaging Notebook, which contains the following information:

1. Date
2. Cooler number (Julian date format)
3. Station number
4. Resealed date
5. Number of paint cans
6. Signed and dated pages

A second log, the Sample Log-in Sheet, is also completed. This log-in sheet contains the following information:

1. Sample custodian's signature
2. Date
3. Batch number
4. Case number
5. Airbill number
6. Storage area
7. Delivering agent's signature
8. Document control number
9. Custody seal - present/absent, intact/not intact
10. Chain-of-custody record - present/absent
11. Sample tags - present/absent
12. Sample tag numbers - listed/not listed on chain-of-custody record
13. Sample Management Office (SMO) forms - present/absent
14. Date received
15. Time received
16. Chain-of-custody record number
17. Client/SMO sample number
18. Corresponding sample tag and laboratory sample numbers
19. Agreement of information on documents
20. Remarks

LCDSS samples are received at ERC-UNLV in shipping coolers containing vermiculite and samples individually sealed in paint cans. There is one eight-ounce sample bottle inside each paint can which is sealed with custody seals and wrapped in plastic zip-lock bags. The paint cans also had custody seals affixed to the outsides of the cans. Chain-of-custody records accompanied each cooler received. Airbills received with the coolers are for each shipment, and one airbill may relate to multiple coolers in the shipment.

All coolers were numbered as they were received, and a record is kept (the unpackaging log, described earlier) to track which samples were shipped in each cooler. Tracking the coolers and samples in this fashion allows the homogenized samples to be shipped for analysis in the same coolers in which they arrived at ERC-UNLV.

Standard operating procedures (SOPs) for sample receiving and the sample custodian's duties and responsibilities are

documented in ERC-UNLV's Chain-of-Custody Standard Operating Procedures (3-21-86). The auditors read these SOPs, and they generally reflected the procedures used for sample receiving for this project.

SAMPLE STORAGE

Samples are stored in sealed shipping coolers located in the laboratory hallway and sample receiving room. The laboratory facility conforms to the requirements of a secure area in that all outside access doors are locked at all times; visitors must sign a visitor log and be escorted while in laboratory areas. The front door of the laboratory is equipped with a code key entry system which may also be operated by the receptionist from her office. All visitors must pass through the reception area prior to entering the laboratory.

Samples stored in the coolers are identified with sample labels containing the following information:

1. Station number
2. Date
3. Comments
4. Time
5. Collector
6. Project number
7. Client
8. Love Canal number
9. Preservative
10. Analysis required

Each homogenized sample is placed into two, four-ounce jars designated A and B. The A jars are placed into archive storage, and the B jars will be sent to Contract Laboratory Program (CLP) laboratories for dioxin analyses. The four-ounce jars are labeled in the same fashion as the eight-ounce jars, and sample tags are also attached to the sample jars.

SOPs for sample storage and security are documented in ERC-UNLV's Laboratory Security Standard Operation Procedures (3/21/86) and Chain-of-Custody Standard Operating Procedures (4/22/86). The auditors read these SOPs, and they generally reflected the sample storage and security procedures used by the ERC-UNLV for this project.

SAMPLE TRACKING

Samples may be tracked through the laboratory from receipt to completion of the homogenization procedure by using the following documents:

1. ERC-UNLV Sample Receipt Log

2. LCDSS Homogenization and Unpackaging Notebook
3. Sample Log-in Sheet
4. Field chain-of-custody records
5. Laboratory chain-of-custody records
6. LCDSS Sample Homogenization Form
7. Preparation Laboratory Notebook
8. Cross-Reference Logbook

The dates of receipt, receipt signatures, and custody and sample sample condition information is recorded on the ERC-UNLV Sample Receipt Log, LCDSS Homogenization and Unpackaging Notebook, and the Sample Log-in Sheets. (These documents were described earlier in the report). The field chain-of-custody records contain the custody transfer dates and signatures, laboratory receipt signatures and dates, sample numbers, and the airbill numbers.

Prior to transferring the samples from the ERC-UNLV facility to CLP laboratories for analysis, and inter-laboratory chain-of-custody record is prepared for each shipment. These custody records contain the following:

1. Name of unit/address
2. Sample number (Love Canal number)
3. Number of containers
4. Sample descriptions
5. Person assuming responsibility (date/time)
6. Sample number
7. Relinquished by
8. Received by
9. Time
10. Date
11. Reason for change of custody

Sample homogenization information is documented, by sample, on the LCDSS Sample Homogenization Forms, Preparation Laboratory Notebooks, and the Cross-Reference Logbooks. These documents contain the information described below:

<u>LCDSS Homogenization Form</u>

PART I

1. Station number
2. Form number
3. Name of mixer
4. Date
5. Time
6. Comments

PART II

7. Sample number
8. Laboratory name (CLP lab destination)

9. Chain-of-custody form number
10. Dioxin shipment record from number
11. Date shipped

PART III (Archive Samples)

12. Sample number
13. Laboratory name (CLP lab destination)
14. Chain-of-custody form number
15. Dioxin shipment record form number
16. Date shipped

PART IV

17. QC sample type
18. Previous blank (Love Canal number)
19. Associated sample number with blank
20. Signature of preparer

Preparation Laboratory Notebook

1. Station number
2. Custody seal condition
3. Cooler ID number
4. Assigned number
5. Reseal date
6. Box number
7. Signature/date

Cross-Reference Log

1. Station number
2. Love Canal number
3. Archive number
4. Hand mix (method)
5. Signature/date

SOPs for sample tracking are currently being developed and will be included in the LCDSS Quality Assurance Project Plan (QAPP). A copy of these procedures was not available for review during the audit. Analytical and quality assurance/quality control procedures are included in the LCDSS QAPP.

DOCUMENT FILING

Project documents are filed by type in project notebooks. These documents will, according to A. Morera, be incorporated into the master project file at CH_2M Hill at the completion of the project.

During the audit, the auditors reviewed all available project documents received by ERC-UNLV for this project. The following deficiencies were identified:

1. Airbills were not signed and dated.

2. SMO forms are not used on this project. The Sample Log-in Sheets should thus be marked "N/A" rather than "absent" for the SMO forms receipt indication.

3. Custody records II-4-5/1038 and II-10-9/1145 need to be signed and dated.

SOPs for document control and organization specify the use of a document control inventory for sample receiving documents. These documents are currently being numbered and inventoried as they are received. Because the documents will become part of the comprehensive LCDSS project file at CH_2M Hill, it is not required that ERC-UNLV number and inventory their project file documents. They are only required to produce their project documents to CH_2M Hill at the conclusion of the project.

SUMMARY

At the conclusion of the audit, a debriefing was held with ERC-UNLV, CH_2M Hill, and Region II personnel. During this debriefing, the REACT auditors made the following comments and recommendations:

1. Chain-of-custody and document control procedures are in place and have been implemented. ERC-UNLV appears to be adhering to these procedures with the following exceptions:

   a. Airbills were not signed and dated.
   b. Custody records II-4-5/1038 and II-10-9/1145 were not signed and dated.
   c. SMO forms are marked "absent" on the Sample Log-in Sheets rather than "N/A" (not applicable).
   d. Sample tracking SOPs were not available for review during the audit. (According to J. Campana and A Morera, these SOPs will be incorporated into the updated QAPP).

2. It was recommended that the airbills accompanying EPA sample shipments be signed and dated upon receipt by the laboratory.

3. It was recommended that all custody records be signed and dated by the laboratory as required.

4. It was recommended that SMO forms be noted "N/A" rather than "absent" on the Sample Log-in Sheets.

APPENDIX C
Quality Assurance Report

Prepared by

U.S. EPA Region II Environmental Services Division
Edison, New Jersey

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION II

DATE: MAR 1 0 1988

SUBJECT: Love Canal Dioxin Soil Sampling Study: QA Report

FROM: Lloyd Kahn, Quality Assurance Officer
Air and Water Section (2ES-MM)

TO: Douglas Gabarini, Project Manager
New York/Caribbean Remedial Action Branch (2ERRD-NYCRA)

Attached is the final report entitled "Quality Assurance Report, Love Canal Dioxin Soil Sampling Study".

I hope that you will find this draft acceptable. Please, call me at FTS 340-6709 if you have any questions.

Attachments

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REGION II FORM 1320-1 (9/85)

QUALITY ASSURANCE REPORT

LOVE CANAL DIOXIN SOIL SAMPLING STUDY

Introduction

The objective of this report is to describe the quality assurance program designed to ensure that the data produced for the Love Canal dioxin soil sampling study were comparable, and were of known accuracy and precision. This report covers the following activities:

1. Development of an approved quality assurance project plan (QAPjP) prior to the initiation of sampling and/or analysis,

2. Evaluation and approval of laboratories capable of handling the analytical requirements and workload in a timely fashion,

3. Submission of performance evaluation samples and matrix blanks,

4. Evaluation of analytical data, and

5. Technical systems audits to verify that field and laboratory personnel adhered to the approved QAPjP and CLP protocols.

Quality Assurance Project Plan Development and Implementation

The QAPjP for the study was developed by the contractor, CH2M Hill, and submitted to the Region II project officer for review and approval. Subsequently, this plan was revised in response to Region II comments. Pertinent parts of the plan dealing with sampling and analysis were approved by the quality assurance officer before sampling was started.

This approval did not include the required homogenization procedure. This part of the QAPjP was developed by the University of Nevada Quality Assurance Laboratory Environmental Research Center (UN-LV) under a contract arrangement with EPA's Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV). The procedure was submitted to the Region II project officer and approved by the quality assurance officer prior to the start of homogenization of the samples. The homogenized samples were then submitted by UN-LV to the laboratories for analysis.

Evaluation and Approval of Laboratories To Perform the Dioxin Analyses

Four CLP laboratories were employed for the routine analysis of 2,3,7,8-TCDD by low resolution GC/MS. These were the Environmental Testing and Certification Laboratory (ETC) of Edison, New Jersey; Eagle Picher of Miami, Oklahoma; Kansas City Scientific Inc. (KCSI) of Kansas City, Missouri; and TMS Analytical Services of Indianapolis, Indiana. The Enseco/California Analytical Laboratory of West Sacramento, California (ECAL) was used to perform additional 2,3,7,8-TCDD analyses under a CLP Special Analytical Services (SAS) contract. However, most of the analyses were conducted by ETC.

At the start of this study, ETC was the only laboratory which had been awarded a contract for the dioxin analysis. The laboratory was audited on January 8, 1987 by Region II personnel to check its capability to perform the dioxin analyses. The team doing the inspection concluded that the laboratory could produce acceptable 2,3,7,8-TCDD data. EMSL-LV performed an audit of ETC on March 20, 1987. The auditors found only minor deficiencies in the laboratory's modus operandum.

EMSL-LV audited Eagle Picher on January 7, KCSI on January 6, TMS on January 8 and ECAL on June 17 - 18, 1987. All laboratories were found to be capable of producing acceptable 2,3,7,8-TCDD data.

As a condition of obtaining a CLP contract, each of the laboratories was required to analyze a performance evaluation sample within the acceptable limits determined by EMSL-LV.

Performance Evaluation Samples and Matrix Blanks.

Performance evaluation (PE) samples are samples prepared by the addition of weighed amounts of an analyte to an environmental sample which did not contain that analyte, or an environmental sample contaminated with the analyte, but for which the analyte concentration was determined by multiple analyses and a mean, or target, value was calculated. For each case, stanmdard deviations are calculated from the multiple analyses and limits for acceptability statistically established. The samples are submitted as unknowns to the laboratories.

For this study, the analyte in the PE samples was 2,3,7,8-TCDD and the samples were either supplied by EMSL-LV or were prepared by an EPA contractor, NUS. The respective PE samples contained target values of 0.8, 1.2 and 1.54 ug/kg 2,3,7,8-TCDD.

A PE sample was submitted with each batch of samples to evaluate whether the laboratory was able to analyze the sample within acceptable limits. A set of data was rejected if the PE sample result was not within the acceptable limits set by EMSL-LV. with each batch of environmental. A reanalysis of the rejected batch was then required.

A matrix blank is a sample of similar physical nature as the environmental samples, but which does not contain a measurable amount of the analyte in question. Two matrix blanks were submitted with each set of samples. The CLP contract requires that each laboratory add a known amount of 2,3,7,8-TCDD to one of the matrix blanks. This is called the matrix spike. The laboratory must then analyze the matrix blank and the matrix spike and calculate the percent recovery of the 2,3,7,8-TCDD. This procedure establishes that a laboratory (1) is not inadvertently contaminating the samples with the analyte and (2) is getting acceptable recoveries of the added 2,3,7,8-TCDD, which indicates that the sample matrix is not interfering with the analysis. An out-of-limits recovery of the matrix spike analyte will not by itself cause data to be rejected, but is an important factor, taken together with the signal-to-noise ratio, method blank and internal standard recovery in evaluating the acceptability of a set of data.

Evaluation of Analytical Data

All analytical data were evaluated by Region II analyst/ reviewers trained in GC/MS analysis and having specialized knowledge in dioxin analysis. The protocol used for the reviews is described in Region II's SOP No. HW-5, "CLP Dioxin (2,3,7,8 TCDD) Data Review", Revision II, dated 12/14/87. This, and its earlier versions, are based upon the requirements of the IFB WA-86-K357 "Statement of Work, Attachment A" for dioxin analysis. The validation of data is based upon the completeness of the deliverables in the CLP contract, proper qualitative and quantitative calibration practices, and the attainment of the criteria for chromatographic column resolution, signal-to-noise ratios, maximum possible concentration, matrix blank and matrix spike recoveries, the percent difference between laboratory duplicates, and PE samples.

Reanalysis, also, was required for each sample for which a laboratory was unable to achieve a "maximum possible concentration" of at least 1.0 ug/kg.

Since data sets were rejected and reanalyzed when the PE sample results were not within acceptable limits, all PE samples accompanying data batches had acceptable 2,3,7,8-TCDD values.

Table I shows the values obtained on duplicate results. Of the duplicates analyzed, only 5 were above detectable limits and showed good repeatability.

Field and Laboratory Audits

The audits performed on the laboratories performing the 2,3,7,8,-TCDD analyses have been discussed above.

In addition, Techlaw, Inc., of Lakewood, Colorado, a contractor employed by EPA'S National Enforcement Investigation Center at Denver, Colorado, did a field audit of the CH2M Hill Love Canal dioxin soil sampling operation on November 17 - 19, 1986 and a laboratory evidence audit of UN-LV in December, 1986.

The auditors concluded that both organizations followed the QAPjP and performed their tasks in an acceptable fashion.

TABLE I

DUPLICATE SAMPLE RESULTS

Sample/Station Number	Original Result ug/kg	Duplicate Result ug/kg
1969	0.04	0.03
2153	0.12	0.11
3199	0.03	0.02
4097	0.12	0.10
4225	0.17	0.17
All Remaining Samples (137)	ND	ND

ND = Not Detected

SOP NO. HW-5

CLP DIOXIN (2,3,7,8 TCDD) DATA REVIEW

REVISION II

PREPARED BY: Stelios Gerazounis DATE: 12/14/87
Stelios Gerazounis
Monitoring Management Branch

CONCURRED BY: Louis Bevilacqua DATE: 12/14/87
Louis Bevilacqua
Monitoring Management Branch

APPROVED BY: Gerard F. McKenna DATE: 12/14/87
Gerard F. McKenna, Chief
Monitoring Management Branch

Date: Dec. 1987
Number: HW-5
Revision: 2

Title: CLP Dioxin Data Review

1.0 Introduction

1.1 This procedure is applicable to dioxin data from contractor laboratories working under the Hazardous Waste Site Contract Labortory Program (CLP).

1.2 The data validation is based upon analytical and quality assurance requirements specified in Solicitation, Offer and Award No. IFB WA-86K357.

2.0 Responsibilities

2.1 The reviewers will complete and/or file the following:

2.1.1 Data Review Log - Each reviewer will fill in the following information in the log book for each data package:

a. date of start of case review
b. date of completion of case review
c. site
d. case number
e. contract laboratory
f. number of samples

2.1.2 Rejection Summary Form - Fill out as necessary using a ratio format. The numerator would indicate the number of dioxin data rejected; the denominator would indicate the number of dioxin fractions containing rejected compounds. File in the appropriate folder.

2.1.3 SMO Report - This form is intended for use in improving the laboratories' performance. Record all reasons for having rejected data along with any contract violations which may or may not have lead to any rejections. Make four copies. Give secretary four copies with instructions to mail to Numbers 1, 2, 3 on the SMO mailing list and to the office corresponding to the region where the lab is located. File the fifth copy in the appropriate folder.

2.1.3.1 In lieu of SMO Report, comments may be appended to end of review narrative under "Contract Problems/Non-Compliance". Do not make copies for above distribution.

2.1.4 Telephone Log - All phone conversations must be initiated by the data review manager or the deputy project officer. If a phone call has been made, fill out stating the bare facts of the conversation. Give two copies to secretary, one to go to the laboratory and one to go to SMO. File the third copy in the appropriate folder.

2.1.5 Data Assessment Checklist - Attachment 1 of this SOP must be completed along with the Data Assessment Narrative.

2.1.6 Forwarded Paperwork - The following are to be forwarded to the Surveillance and Monitoring Branch (SMB) upon completion of the review:

a. data package
b. completed data assessment checklist (original)
c. Contract Compliance Screening (CCS)
d. CLP Re-analysis Request/Approval Record (original and 2 copies)

2.1.7 Filed Paperwork - The following are to be filed within MMB files:

a. SMB Review (Copy)
b. Narrative part (Para. 6.0) of data assessment checklist (copy)
c. SMO Report (original)
c. Telephone Record Log (yellow copy)
d. Rejection Summary Form (original)
e. CLP Re-analysis Request/Approval Record (copy)

3.0 Data Completeness

Incomplete data packages must be brought to the attention of the data review manager or the deputy project officer (DPO) whenever the lack of any information would cause the rejection of data.

4.0 Rejection of Data

All values determined to be unacceptable on the dioxin analysis data sheets should be lined over with a red pencil.

5.0 Acceptance Criteria

In order that reviews be consistent among reviewers, acceptance criteria as stated in Attachment 1 should be used. Additional guidance may be found in National SOP for data review.

6.0 Reviewer Corrections

All corrections to reviewed data made by reviewers must be indicated in red pencil.

7.0 SMO Contract Compliance Screening (CCS) and FIT Preliminary Review

This is intended to aid reviewer in locating any problems, both corrected and uncorrected.

8.0 Request for Reanalysis

Data reviewers must note all items of contract non-compliance within Data Assessment Narrative (see page 15 of this SOP). If holding times and sample storage times have not been exceeded, the Data Review Manager or DPO may request reanalysis, if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record".

SURVEILLANCE AND MONITORING BRANCH REVIEW

Project Name/Site:_____

Case Number:_____

Type Investigation (Circle One): . Remedial Site Other_____

Contract No.:_____

Laboratory:_____

Sample Identification Numbers:

Aqueous:_____

_____

_____

Soil/Sediment:_____

_____

_____

Superfund Account No.:_____

Comments:_____

_____

_____

_____

_____

_____

NOTE TO THE REVIEWER: The attached SOP is for a "Total" Review of the 2,3,7,8-TCDD data. Its purpose is to facilitate the validation of the data reported by the contracting laboratory and also to assure that the data is being reviewed in a uniform manner.

Please read each item carefully and check YES if there is compliance and NO if there is no compliance.

The reviewer must red-line unacceptable data on Sample Data Sheets (red-lined does not imply the compound is not present).

Red-lined or rejected data means that the data is unusable and that the sample may need to be reanalyzed by the same laboratory. If the laboratory already has analyzed the sample twice or the sample cannot be analyzed by the method, a reanalysis must be done by another laboratory.

Please notify immediately the Deputy Project Officer (DPO) or the Data Review Manager concerning problems and inadequacies with regard to laboratory sample data.

1.0 Deliverables

1.1 All deliverables must be clearly labeled with the SMO case number and the associated sample/traffic number. Review the data package to assure that all items listed below are provided. Missing, illegible or incorrectly labeled items must be checked off. The contractor should immediately be contacted and requested to submit the missing or incorrect items.

1.2 Are the following forms present?	YES	NO
a. TCDD Data Report Form (Form B-1)	___	___
b. Initial Calibration Summary (Form B-2)	___	___
c. Continuing Calibration Summary (Form B-3)	___	___
d. A Chronological List of all Sample Analyses	___	___
GC/MS Displays		
a. SICP's for the Initial Triplicate Analysis of the Four Concentration Calibration Solution	___	___
b. SICP for Each Performance Check Solution	___	___
c. SICP for Each Shift Standard	___	___

	YES	NO
GC/MS Displays		
d. Plotted Concentration Calibration Curve	___	___
e. SICP's for Each Sample Ran	___	___

WERE THE FOLLOWING CRITERIA MET?

2.0 Instrument Performance

2.1 Performance Check Solution Requirements - The performance check solution must be analyzed at the beginning and at the end of each 12 hour shift. ___ ___

2.1.1 Chromatographic Resolution

- The valley between 2,3,7,8-TCDD and the peak of all other TCDD isomers must be $\leq$ 25%. ___ ___

- Recalculate the valley (%) between 2,3,7,8-TCDD and the peak of the least resolved adjacent isomer using the following equation:

$$\% \text{ Valley} = \frac{x}{y} \times 100$$

Where: y = peak height of 2,3,7,8-TCDD
x = measured height from valley of the least resolved adjacent isomer to the baseline.

2.1.2 Ion Critera

- The ratio of integrated ion current for m/z 320 to m/z 322 for 2,3,7,8-TCDD must be $\geq$.67 and $\leq$.90. ___ ___

- The ratio of the integrated ion current for m/z 332 to m/z 334 for $^{13}C_{12}$2,3,7,8-TCDD must be $\geq$.67 and $\leq$.90. ___ ___

ACTION: 1) If the 25% valley requirement is not met, red-line all data collected within the shift associated with the performance standard. No action is taken if any of the other requirements are not met; and 2) if the performance check solution was not analyzed in the required frequency, use professional judgment to determine the effect on the quality of the data.

	YES	NO

3.0 Calibration

3.1 Initial Calibration - Initially, 2,3,7,8-TCDD Standards of 0.2, 1.0, 5.0, and 20.0 ng/ul solutions will be injected in triplicate. Each solution also contains 0.028 ng/ul of $^{37}Cl_4$2,3,7,8-TCDD (surrogate), 1ug/ul $^{13}C_{12}$2,3,7,8-TCDD (internal standard) and 0.6ng/ul $^{13}C_{12}$1,2,3,4-TCDD (recovery standard). The concentration of these solutions may vary only slightly from time to time. The calibration standards must be analyzed using the same GC conditions that were used to analyze the performance check solution and must meet the following criteria.

- Scanning time must be $\leq$ 1.5 seconds.	___	___
- The ratio of the integrated ion current for m/z 320 to 322 for 2,3,7,8-TCDD must be $\geq$.67 and $\geq$.90.	___	___
- The ratio of integrated ion current for m/z 332 to m/z 334 for $^{13}C_{12}$2,3,7,8-TCDD and $^{13}C_{12}$1,2,3,4-TCDD must be $\geq$0.67 and $\leq$0.09.	___	___
- The signal to noise ratio for ions 332 and 334 must be $\geq$ 10.	___	___
- The signal to noise ratio for ions 259, 320, 322, and 328 must be $\geq$ 2.5.	___	___
- The ions 259, 320, and 322 must maximize simultaneously and within 3 seconds of ions 332, 334 of $^{13}C_{12}$-2,3,7,8-TCDD.	___	___

For each concentration level, recalculate the response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD:

$$RRF_n = \frac{Ax \times Qis}{Ais \times Qx}$$

Where: Ax = of the sum of integrated ion abundance of m/z 320 and m/z 322 for unlabeled 2,3,7,8-TCDD.

Ais = the sum of integrated abundance of m/z 332 and m/z 334 for $^{13}C_{12}$-2,3,7,8-TCDD

Qx = quantity of unlabeled 2,3,7,8-TCDD

Qis = quantity of $^{13}C_{12}$-2,3,7,8-TCDD

ReCalculate the response factor for $^{13}C_{12}$-2,3,7,8-TCDD relative to $^{13}C_{12}$-1,2,3,4,-TCDD:

$$RRF_1 = \frac{Ais \times Qrs}{Ars \times Qis}$$

Where Ars = the sum of integrated abundance of M/Z 332 and M/Z 334 for $^{13}C_{12}$-1,2,3,4 - TCDD

Qrs = the quantity of $^{13}C_{12}$-1,2,3,4 - TCDD.

	YES	NO
- The variation of the RRF for unlabeled 2,3,7,8-TCDD at each concentration level must not exceed 10% RSD	___	___
- The RSD of the 4 - mean RRFs for unlabeled 2,3,7,8-TCDD must not exceed 10% RSD.	___	___
- The RSD of the 4 - mean RRFs for $^{13}C_{12}$2,3,7,8-TCDD must not exceed 10% RSD.	___	___
- The mean of the 4 - mean RRFs for each compound established in the initial calibration must be used for concentration calculations.	___	___

ACTION: 1) If the required initial calibration data were not supplied, notify the laboratory. If they are unavailable, red-line (reject) all data; and 2) if any of the calibration curve standards fail to meet any of the above acceptance criteria red-line all data associated with the calibration curve.

3.2 Calibration Check Standard - At the beginning of every 12-hour shift, a calibration standard of a concentration of 1ppb must be analyzed using the same GC conditions that were used to analyze the performance check standard. Were the following operating MS conditions met?

- Scanning time must be $\leq$ 1.5 seconds.	___	___
- The ratio of the integrated ion current for m/z 320 to 322 for 2,3,7,8-TCDD must be $\geq$.67 and $\leq$.90.	___	___
- The ratio of the integrated ion current for m/z 332 to 334 for $^{13}C_{12}$-2,3,7,8-TCDD must be $\geq$.67 and $\leq$.90.	___	___
- The ions 259, 320, 322 for 2,3,7,8-TCDD must maximize simultaneously and within 3 seconds of $^{13}C_{12}$-2,3,7,8-TCDD.	___	___

	YES	NO
- The signal to noise ratios for ions 259, 320, 322, and 328 must be $\geq$ 2.5.	___	___
- The signal to noise ratios for ions 332 and 334 must be $\geq$ 10.	___	___
Check calculations for the response factors for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD and for $^{13}C_{12}$-2,3,7,8-TCDD relative to $^{13}C_{12}$-1,2,3,4-TCDD.		
- Are there any errors?	___	___
- The response factor of 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD must be within $\pm$ 10% of the mean value for the initial 4-point calibration curve.	___	___
- The response factor of $^{13}C_{12}$-2,3,7,8-TCDD relative to $^{13}C_{12}$-1,2,3,4-TCDD must be within $\pm$ 10% mean value of the initial 4 point calibration curve.	___	___

ACTION: 1) If the required number of analyses of calibration check solutions were not submitted notify the contracting laboratory. If they are unavailable, reject all data without an associated calibration check standard; and 2) if the calibration check standard fails to meet any of the acceptance criteria, reject all the data associated with this standard.

4.0 Sample Data

4.1 Qualitative Requirements

4.1.1 The RT of 2,3,7,8-TCDD must be within 3 seconds of the RT of the $^{13}C_{12}$-2,3,7,8-TCDD.	___	___
4.1.2 The integrated ion currents for m/z 259, 320 and 322 must maximize simultaneously.	___	___
4.1.3 The integrated ion currents for m/z 259, 320, 322, and 328 must be at least 2.5 times background noise and must not have saturated the detector.	___	___
4.1.4 The internal standard ions m/z 332, 334 must be at least 10 times of noise level.	___	___
4.1.5 The ratio of the integrated ion currents m/z 320 to m/z 322 and m/z 332 to m/z 334 must be $\geq$.67 and $\leq$.90.	___	___

	YES	NO
4.1.6 When 2,3,7,8-TCDD is not present, the ratio of internal standard ion m/z 332 to m/z 334 must still be $\geq$.67 and $\leq$.90.	___	___
4.1.7 If the 2,3,7,8-TCDD was not qualitatively identified in the sample, the MPC (detection limits) must not exceed 1 ppb.	___	___

ACTION: 1) if the 2,3,7,8-TCDD qualitative criteria were not met, 2,3,7,8-TCDD was not qualitatively identified, red-line positive value; 2) if the detection limit exceeds 1 ppb, red-line ND (not detected) dioxin value; 3) if the signal to noise criteria for ions m/z 328, 332, 334 were not met, red-line positive or ND dioxin values; and 4) if the ratio of ion m/z 332 to m/z 334 is outside the acceptance criteria, red-line positive or ND dioxin data.

4.2 Quantitative Requirements

4.2.1 Recalculate the concentration of 2,3,4,7-TCDD which was found in the sample using the equation:

$$Cx = \frac{Ax \times Qis}{Ais \times RRF_n \times W}$$

Where: Cx = the concentration of 2,3,7,8-TCDD in micrograms/Kg

Ax = the sum of integrated ion abundance for M/Z 320 and M/Z 322 for unlabeled 2,3,7,8-TCDD

Ais = the sum of integrated ion abundances for M/Z 332 and M/Z 334 which are the characteristic ions for the internal stanard $^{13}C_{12}$-2,3,7,8-TCDD

Qis = quantity of the internal standard (in nanograms) added to the sample before extraction

RRF_n = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to $^{13}C_{12}$-2,3,7,8-TCDD

W = weight (in grams) of wet soil or sediment sample

YES NO

4.2.2 Recalculate the concentration of the internal standard $^{13}C_{12}$-2,3,7,8-TCDD using the formula:

$$Cis = \frac{Ais \times Qrs}{Ars \times RRF_i \times W}$$

Where: Cis = the concentration of the $^{13}C_{12}$-2,3,7,8-TCDD in ug/kg or ug/L

Ais = the sum of integrated ion abundance for M/Z 332 and 334 for $^{13}C_{12}$-2,3,7,8,-TCDD

Ars = sum of integrated ion abundances for M/Z 332 and 334 for $^{13}C_{12}$-1,2,3,4-TCDD

Qrs = quantity (in ng) of $^{13}C_{12}$-1,2,3,4-TCDD added to the sample before injection

RRF_i = calculated mean response factor for $^{13}C_{12}$-2,3,7,8 -TCDD relative to $^{13}C_{12}$-1,2,3,4-TCDD

W = weight (in grams) of wet soil or sediment sample or volume of water extracted (in mL)

4.2.3 The recovery of the internal standard must be within the 40 and 120 percent recovery window. ___ ___

ACTION: These are advisory limits. View outliers in conjunction with surrogate recoveries and other data.

4.3 Recalculate Maximum Possible Concentration - When no unlabeled Dioxin is detected, the lab must calculate the estimated detection limits which is the concentrations required to produce a signal with an area or peak height 2.5 times the background signal area or peak height. To recalculate use the formula:

$$MPC = \frac{2.5 \times Ax2 \times Qis}{Ais2 \times RRF_n \times W}$$

Where: MPC = Estimated maximum possible concentration for unlabeled 2,3,7,8-TCDD in ug/kg or ug/L.

Ax2 = peak height or integrated ion abundance for M/Z 320 or 322

Ais2 = integrated ion abundance or peak height for M/Z 332 when M/Z 320 is used or M/Z 334 when M/Z 322 is used to determine Ax

Qis = same definition as in 4.2.1

RRF_n = calculated mean response factor for 2,3,7,8 -TCDD relative to $^{13}C_{12}$ - 2,3,7,8 - TCDD

W = same definition as above

	YES	NO

NOTE: The use of the area or peak height for M/Z 320 is preferred to M/Z 322 except when there is interference for M/Z 320 but not for M/Z 322

ACTION: If variation in the calculations are found between the contractor's results and the regions recalculated results, the contractor must be informed and the discrepancy resolved. If the calculated amount of unlabeled Dioxin is outside the linear range obtained by the initial calculation (100 ppb for the 4 points) reject the data.

4.4 Estimated Maximum Possible Concentration - For samples where interference is observed for both M/Z 320 and 322 or when an unacceptable ratio prevented identification of unlabeled 2,3,7,8-TCDD, use equation in Section 4.2.1 to estimate the maximum concentration that could be represented by detected signals.

5.0 Quality Control

5.1 Blanks - Field Blank/Reagent Blank

5.1.1 One field blank must be extracted and analyzed for each batch of samples	___	___
5.1.2 One method blank must be extracted and analyzed for each batch of samples or each time a group of samples are extracted.	___	___
5.1.3 Acceptable laboratory method blanks must not contain any signal at 320, 322, or 259 which is greater than 2% of the m/z 332 response within ±5 scans of the m/z 332 peak maximum. If the method blank that was extracted along with a batch of samples is contaminated, the associated positive samples must be rerun. (See Exhibit C.)	___	___

	YES	NO

ACTION: 1) If the proper number of reagent blanks, were not submitted notify the contractor. If they are unavailable, reject all positive sample data; 2) if the field blank is missing, notify the DPO and discuss the action to be taken; 3) if the contamination of Dioxin in the reagent blank or in the field blank is >.1 ppb all positive dioxin data must be rejected; and the sample results <10 times the blank contamination. 4) if the contamination of Dioxin in the blank is <.1 ppb no action is taken; and, 5) if the detection limit in the blank exceeds 1 ppb red-line positive data in all samples associated with the blank.

5.1.4 Matrix Spikes Requirements - Each batch of samples must contain a sample of uncontaminated soil/sediment which is fortified before analysis with a quantity of 1 ug/kg of unlabeled Dioxin, 50 ng of $^{13}C_{12}$-2,3,7,8-TCDD and 10 ng of $^{37}Cl_4$-2,3,7,8-TCDD.

5.1.4.1 Is the percent recovery of 2,3,7,8-TCDD within the 60 to 140 percent range? ___ ___

ACTION: If the recovery is outside the 60 to 140 percent range, no action is taken in regard to the acceptability of the data. It may be used, however, as a criterion to decide questionable cases. The contracting lab must be notified, however, and the problem discussed.

5.1.5 Duplicate Analysis Sample

5.1.5.1 For every batch of samples, is there a sample designated as duplicate? ___ ___

5.1.5.2 Results of laboratory duplicates must agree within 50% relative difference. ___ ___

ACTION: No action is taken if relative difference is >50%. The contracting laboratory must be notified, however, and the problem discussed.

	YES	NO

5.1.6 Performance Evaluation (PE) Sample - A performance evaluation sample which is provided by the region must be analyzed with every batch of sample.

5.1.6.1 Do the analytical results fall within the 99% EMS-LV acceptance criteria? ___ ___

Sigma Code	Target ppb	n	$\bar{x}$	s	Windows 90%	Windows 95%	Windows 99%
14	7.7	11	5.7	0.92	(3.9-7.4)	(3.5-7.8)	(2.6-8.7)
16	10.0	8	6.7	0.61	(5.4-7.9)	(5.1-8.2)	(4.4-8.9)
17	5.0	23	4.1	0.54	(3.1-5.0)	(2.9-5.2)	(2.5-5.6)
18	3.3	48	3.4	0.57	(2.4-4.3)	(2.2-4.5)	(1.8-4.9)
24	1.0	0	---	----	---	---	---
25	1.0	44	0.84	0.16	(0.56-1.1)	(0.51-1.2)	(0.40-1.3)
26	10.0	57	7.9	0.66	(6.8-9.0)	(6.6-9.2)	(6.2-9.6)
—	---	00	1.5448	0.22176		(1.10-2.00)	(0.88-221)
--	---	25	1.5448	0.22176		(1.09-2.00)	(0.72-2.37)

ACTION: 1) The PE raw data must be reviewed as if it were an environmental sample; 2) if 2,3,7,8-TCDD was not qualitatively identified reject all data; 3) if the reported value for PE sample exceeds the upper acceptance limits, reject all positive Dioxin values in the entire associated batch of samples, but not the ND values; and 4) if the reported value exceeds the lower acceptance limits, all positive and negative (ND) values are rejected.

6.0 Holding Times (For water sample only).

6.1 Have any holding times have been exceeded for aqueous samples

a. 10 days to extraction from verified time of sample receipt ___ ___

b. 40 days after extraction for analysis ___ ___

Action: If these holding times are exceeded, red-lined the value on the data sheet (including detedtion limits) indicating rejection.

CASE#_____ LAB_____ Site_____

7.0 Conclusions: (NOTE: Reviewers must red-line unacceptable data on sample data sheets; red-line data does not imply the compound is not present)

7.1 Data Assessment Narrative_____

_____

_____

_____

_____

_____

_____

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_____

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_____

_____

7.2 Contract Problems/Non-compliance _____

_____

_____

_____

_____

Reviewer's Signature: _____ Date:_____

Verified By: _____ Date:_____